COMPRESSION OF FOODS DURING VACUUM FREEZE DEHYDRATION

by

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ABSTRACT

Reversible reduction in volume of freeze-dried food so that its dry density approaches that of fresh product will result in significant efficiencies, especially in the areas of package material requirements, storage, and transportation.

The technique used in this study is simultaneous compression of the food material by mechanical means during the "normal" course of freeze-drying process to produce low-moisture, high density product.

Compression of foods during freeze drying can be considered as an efficient method, since it minimizes the number of processing steps (i.e., initial overdrying, humidification and final redrying that are required in currently practiced methods).

Investigation of the influence of several process parameters on the degree of compression showed that the applied compressive pressure is the main factor determining the final degree of compression. It was noted that increasing the applied pressure also acted to accelerate the drying process, this being attributed to improved thermal conductivity and heat transfer through the already dried, compressed layer.

As expected, increasing the level of heat input accelerates the freeze-drying process. However, within limits it did not have any apparent effect on the final degree of compression.

The size of the sample cell holders used and porosity of the sample holder material used in this study did not seem to have any significant effects on drying and compression behavior of the product.

This study has included an investigation of the compression behavior of freeze-dried green beans rewetted to moisture levels between 6% and 45% (dry solid basis) at temperatures of -25°C to +24°C, using the Instron Universal Testing Machine. The results showed that both moisture and temperature strongly affect the mechanical properties of the food. Within the range of moisture and temperature investigated, the effect of increasing either or both of these variables can be described as a decrease in the relative rigidity of the food structure, this being indicated by the level of the stresses that are required to reach a given strain and variation of the values of the modulus.

It was further shown that the effects of moisture and temperature are inversely interactive so that there exist specific combinations of moisture content and temperature which correspond to common values for mechanical properties and result in a similar compression response under stress. This indicates that given either a value of moisture and temperature or a value of a pertinent mechanical property, it is possible to predict and construct a stress-strain relationship for freeze-dried green beans.

From evaluation of literature information on moisture and temperature gradients that exist in the dry layer during freeze drying, it was shown that the resulting values of mechanical properties are suitable for compression during freeze drying. These results indicated that the same factors (moisture content and temperature) which govern the compressive behavior of food in the objective testing method using the Instron Universal Testing Machine also govern the compression behavior during freeze drying.

The correlations of compressive behavior obtained from the Instron studies also were used to evaluate the results obtained in compression during freeze drying. Based on these correlations, a simple method was developed for the prediction of the compression behavior of the foods during freeze drying.

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1. INTRODUCTION

Freeze drying has long been recognized as the method of dehydration giving products with high quality and retained nutritional value. Freeze-dried foods can be quickly rehydrated and when rehydrated they closely resemble the food prior to dehydration. When foods undergo freeze drying, however, there is very little volume reduction from the original food form.

While the retention of physical shape has long been viewed as desirable since it indicated no structural change in the product, it does result in an open and porous structure which gives a very light, fragile product of low packaging density. The disadvantages related to low packaging density can be summerized as:

- The open structure is susceptible to oxidation and moisture pickup, and thus special packaging is often required.
- Large package volumes are required to enclose small product weights.
- Readily friable products produce fines during handling and transportation which is essentially a product wastage.
- Large storage space is required.

Under most situations, it becomes extremely advantageous to compact dehydrated foods by reducing or eliminating the void volumes which exist between individual pieces and the internal void space which was occupied by water prior to dehydration. For such compacted or compressed foods, there are greatly reduced packaging, handling, transportation, and storage requirements which gives a significant potential economic savings. The requirements made on the compressed food products are that upon rehydration they regain their original geometry and resemble their non-compressed counterparts in color, odor, flavor, texture, and appearance.

To avoid fragmentation and irreversible structural changes the product must be in a compressible state during the compression process.

The currently practiced method for producing freeze-dried and compressed foods has been described by Rahman et al. (1969). In short, the product is pretreated as required, blast frozen and freeze dried to a final moisture content of less than 2%. The freeze-dried product is then made compressible by either thermal treatment (Rahman et al., 1970) or by addition of low-molecular-weight agents such as water (Hamdy, 1962; Ishler, 1965) or glycerine, glycerol, propylene glycol (Brockman, 1966; Rahman et al., 1969). Water is considered to be the most desirable agent since it imparts no foreign flavor to the rehydrated product, and the current industrial practices for addition of moisture to freeze-dried products prior to compression include steaming and water misting (Rahman, personal communication). In both rewetting processes, the freeze-dried foods are brought to 5% to 15% moisture.

Compression is accomplished uniaxially at pressures between 250 to 2000 psi after which the product is redried to a moisture content of less than 2% and packaged.

The overdrying, rewetting, and subsequent redrying of the product presents a major inefficiency in the process, and studies on other methods for preparing freeze-dried/compressed foods have been initiated such as "limited freeze drying" and "partial freeze drying and microwave treatment."

The studies to be reported on herein have considered an approach where compression and drying of the food material to a low-moisture, high-density product is accomplished simultaneously and in a single-step process.

In this process,when food is under stress during freeze drying, it undergoes a reduction in volume as well as weight which can be later recovered by rehydration of the food.

Figure 1-1 shows the different methods that have been used to produce freeze-dried/compressed foods.

Included in this, there are studies of the mechanical properties of the food which relate to the compression behavior during the freeze-drying process.

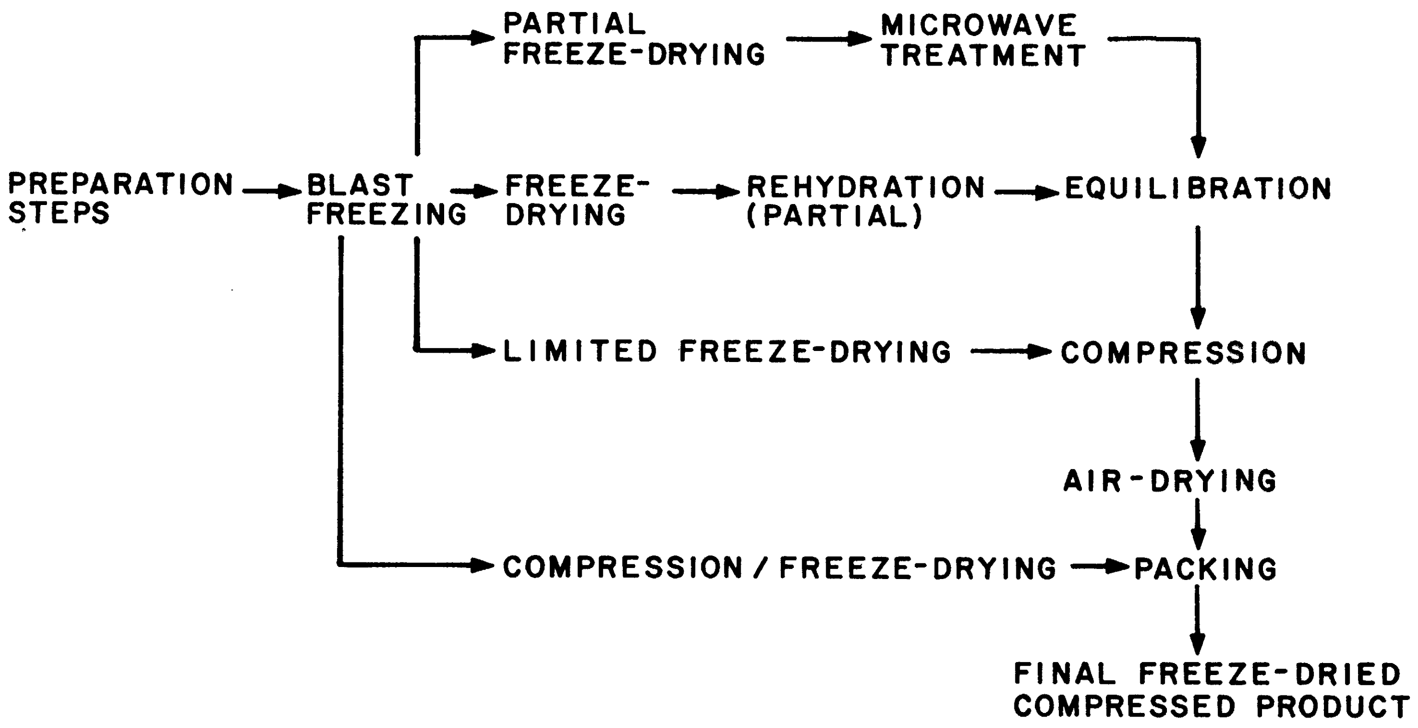


Fig. 1-1 Methods of preparation of freeze-dried/compressed products.

2. LITERATURE SURVEY

2.1 Freeze Drying

Vacuum freeze drying is a method of dehydration in which water is removed from foods by transfer from the solid state (ice) to the gaseous state (water vapor). This sublimation process can be accomplished by reducing the temperature and vapor pressure of water below the triple point (about 0°C and 4.5 torr) and by supplying the energy of sublimation (about 600 cal/gm). This can be achieved by (1) radiation to the outer dry layer of the drying food, (2) conduction through the frozen layer, or (3) internal heat generation (microwaves). Lastly, it is necessary to remove the water vapor generated by sublimation with a moisture sink (King, 1971).

Advantages of this technique include the low processing temperature, the relative absence of liquid water and rapid transition of any local region of the material from a fully hydrated to a nearly completely dehydrated state. This rapid transition minimizes the extent of various degradative reactions which often occur during drying. The low temperatures involved also help to minimize these reactions. The absence of the liquid state further helps to minimize degradative reactions and prevents surface concentration of solutes leading to poor rehydration ability (King, 1971).

Despite the fact that freeze drying gives dehydrated products of very high quality and high retained nutritional value, it involves high initial capital investment, high processing costs, for some products it requires special treatment (cutting, slicing, etc.) prior to drying to improve dehydration, and results in products of very low density (Longmore, 1973).

2.2 State of Water and Solutes in Frozen and Freeze-Dried Systems

The most important change that occurs during freezing is the appearance of at least one new phase (i.e., conversion of water into ice). According to Mackenzie (1965), other changes include:

- Dehydration of the insoluble solid material
- Coalescence of droplets of immiscible liquids
- Crystallization of some solutes
- Formation of amorphous precipitates of other solutes
- Exclusion of dissolved gases
- Formation of concentrated solute glasses
- Disruption of molecular complexes (lipo-proteins)

The ideal frozen state would show a sharp and discrete dividing surface between a region which consists of ice crystals and a region which consists of concentrated amorphous solution. However, there is substantial evidence that of the total water present in food, a portion is strongly bound to individual sites of the solute components, while an additional amount is bound less firmly, but still is not available as solvent for various food components (Karel, 1974).

It has been established that upon cooling water-containing foods to low temperatures, well below the freezing point of the food, a portion of the total water remains unfrozen. This "unfreezable water," which is estimated to be in the range of 0.2 - 0.4 g per g food solids is less free than liquid water but has greater mobility than ice. The portion of water which is held more strongly than "unfreezable water" is called "monolayer value" and is assumed to be bound to specific sites in foods. Foods contain many polar sites including, for instance, the hydroxyl groups of polysaccharides, carbonyl and amino groups of proteins, and others to which water can be held by hydrogen bonding, ion-dipole bonds or by other strong interactions (Karel, 1974). The monolayer value is well below the unfreezable water content, it being estimated to be 20%-50% of the non-freezing fraction of water (Duckworth, 1972).

2.3 State of Water during Freeze Drying

During freeze drying, the ice region retreats inward as a front, leaving the dried matrix behind. The resultant structure consists of pores (the location of former ice crystals) passing through a dry matrix of insoluble components and precipitated compounds which were originally in solution. It should be noted that if during freeze drying the temperature and moisture content in any region surpasses a critical level, mobility in the concentrated aqueous solution may be sufficiently high to result in flow and loss of the original structure (melting or collapse) and loss of the separation of solutes which existed immediately following freezing (Karel, 1975).

Most analyses of freeze drying kinetics have been based on an assumption of a sharp and discrete dividing surface between a region which is fully hydrated and frozen, and a region which is nearly completely dry. However, in recent years there has been considerable discussion regarding the degree of sharpness of this surface when it is retreating as a frozen front during freeze drying.

2.3.1 Evidence for a Sharp Sublimation Front

King (1971) explains that a very sharp front would result if sublimation occurred from a very thin zone near the surface of the frozen region, and if the sublimation removed essentially all of the initial moisture from the remaining solid material immediately after the passage of the frozen front from that region.

Many investigators have reported cutting open partially freeze-dried specimens and observing a distinct and relatively sharp demarcation between the still frozen zone and dry zone of uniform coloration. These include Hardin (1965) who studied freeze-dried beef, Margarities and King (1969) who studied freeze drying turkey meat and Beke (1969) who studied freeze drying of pork.

Additional evidence is obtained by Hatcher (1964) who used gamma ray attenuation measurements to monitor the profile of moisture content across relatively thick (2 inches) slabs of beef as a function of time during freeze drying. Hatcher reports that visual inspection of partially dried specimens revealed the ice front to be planar. Since his gamma ray beam diameter was 3/16 inch, he concluded that the ice front was sharper than could be detected by the beam. He also found that the residual moisture in the dry zone was so low that it did not affect the gamma ray count rate.

MacKenzie (1966) constructed a "freeze drying microscope" in which he could observe freeze drying as it occurred in various transparent frozen liquid systems. An extremely sharp sublimation front was observed, although the front, while sharp, did not necessarily remain planar. There should be a finite partial pressure of water vapor in the gas within the dry layer, since the water vapor generated by sublimation must escape across the dry layer. Therefore it should be expected that a finite amount of sorbed water will be present in the dry layer corresponding to at least the amount that would be predicted by equilibrium sorption isotherm. Sandall (1966) made calculations for typical conditions of freeze drying of turkey breast meat using the sorption isotherm for the same material that was measured by King et al. (1968). For heating from the outer dry surface, he concluded that equilibrium between the dry layer and the escaping water vapor would give an average moisture content within the dry layer equal to 3 percent of the initial water, or about 1.5 percent moisture content on a solid basis. However, this surprisingly low value seems to result from the increasing temperatures across the dry layer toward the outer surface as well as from the decreasing water vapor partial pressure (King, 1971).

2.3.2 Evidence for a Diffused Sublimation Front

Meffert (1963) measured temperature profiles during freeze drying of rutabaga. From the temperature and thermal conductivities he inferred from them, it was concluded that some 30 to 40 percent of the initial water content was left behind by the retreating ice front. This remaining water had to be removed by secondary drying or desorption. In 1965 Meffert determined moisture contents in layers of suede, and found basically the characteristic two-zone phenomenon, a dried layer and an ice core. Also, clearly distinguishable was the existence of a moisture gradient through the dried layer that varied from around 3.0 g H_2O/100 g solids near the outer surface to 33.0 g H_2O/100 g solids near the interface when the ice core had receded half way into a 3.6 cm sample dried from both sides.

Brajnikov et al. (1969) interpreted that the results of some experiments demonstrated the presence of a diffuse sublimation front. He took color photographs of cut specimens of beef which had been freeze-dried once, rehydrated with a solution of $CoCl_2$ and then partially freeze-dried again. Divalent cobalt has the property of changing color from pink to blue depending on water activity of its surroundings. Brajnikov's experiments with $CoCl_2$ indicated the presence of a transition zone; however, King (1971) has noted that the cobalt salt can lower the freezing point which could cause the presence of some liquid at the sublimation front, even at -20°C.

Luikov (1969) observed that sublimation and ablimation (desublimation from vapor state to solid state) occur simultaneously at the sublimation front of an ice sphere. The tendency for ablimation to occur depends upon the degree of water vapor saturation of surrounding gas phase. Ablimation occurred to a greater extent as the surrounding gas became more nearly saturated in water vapor.

The degree of sharpness of the ice front during freeze drying reflects the extent of freeze drying and the condition(s) existing during freeze drying. Important parameters which seem directly related to broadening of the ice front are pressure, and temperature difference between local temperature in the "dry" layer and ice front temperature.

Luyet (1962) in describing drying phenomena includes a "pseudo freeze drying" or "secondary drying" stage which accounts for the withdrawal of the non-frozen water. MacKenzie and Luyet (1964) observed with a freeze drying microscope that the secondary drying of the outermost portions of a sample proceeds simultaneously with sublimation of ice from the sample interior.

Bralsford (1967) described secondary drying as taking place at the interface between the dried and frozen region. In his description the interface had a finite thickness which increased as the drying front receded toward the interior of the specimen. He postulated the existence of a "diffusion zone" whose broadening as the zone recedes into the sample was due to solute migration which gives more and more ice being melted. He felt that at higher chamber pressures more liquid water is present in the ice core which results in some liquid diffusion into the dry layer.

Aguilera and Flink (1974) calculated moisture profiles from temperature measurements obtained during freeze drying. Their calculation was based on a relationship originally noted by Gentzler and Schmidt (1973) between the bound water content at a position in the dry layer (W_B) and temperature difference (ΔT_B) between that position in the dry layer (desorption temperature) and the sublimation temperature of ice during the freeze drying. When the mathematical expression is based on higher desorption temperatures for bound water as compared with the sublimation temperature for frozen water, it can be expressed as:

$$W_B = e^{(a\Delta T_B + b)}$$

where a and b are constants dependent on each particular product. Aguilera and Flink (1974) indicated that combining the measured temperature profiles with the above relationship results in higher moisture contents being calculated at a given distance from the ice front as drying proceeds. Their results show that at a heater temperature of 128°C and a surface temperature of 56°C, the diffusion zone was about 2 mm when the ice front had retreated 5mm (after 2.5 hr), while it had expanded to 5 mm thick when the ice had retreated about 15 mm (after 9 hr).

While quantified drying theories, which have assumed a sharp and uniformly retreating ice front (URIF) which leaves behind an essentially dry layer, do not seem to result in large errors in predicting drying times for various materials, it nevertheless seems clear that the existence of a sharp sublimation front is an idealization (Karel, 1975).

2.4 Compression; A Rheological Approach

2.4.1 Compressive Stress

Stress has been defined (Mohsenin, 1968) as the aggregates of all the tractions (force per unit area) corresponding to all directions passing through a point in the body. Complete specification of stress requires the use of at least six "components of stress." Compressive stress is one of the six components of stress. It expresses the linear, uniaxial application of force which causes compaction type volumetric change only. In this text the usage of the term stress refers to compressive stress unless specified.

2.4.2 Degree of Compression

Degree of compression represents the change in volume of a body relative to its original, precompressed volume. It can be expressed numerically in a number of ways, and its numerical value will depend on the method of measuring or defining volume (Emami et al., 1979).

A "displacement" type change considers only the volume of individual sample pieces; no void volume of the bulk sample is considered. Thus, a "displacement" change measure represents the true volume change of a sample piece. Since, in compression of freeze-dried foods, voids that are produced by sublimation of ice crystals are reduced or eliminated, the maximum degree of compression of an individual piece is related to its initial water content. The technique used for determining the volume change of a sample piece is similar to the technique for measuring loaf volume of bread using rape seeds. When the degree of compression is measured by the change in initial and final volumes of the sample pieces when orderly "packed" in the sample holder, the void volumes between the food pieces are also included in the measurement. This is also true for when the food pieces are "randomly packed," for which the initial volume is defined as the volume which is occupied by the given sample when randomly packed in their normal container or package. The randomly packed volume is much larger than when the sample is orderly packed in the sample holder, due to larger void volumes between the sample. Values given in literature for degrees of compression are usually based on randomly packed volumes (Emami et al., 1976).

2.4.2.1 Compressive Strain

Strain has been defined (Mohsenin, 1968) as the relative change in linear dimension, or shape, or volume of an element within a body. Strain, like stress, can be resolved into six components of normal and tangential strains. Compressive strain is one of the normal strains referring to linear, uniaxial compaction type change in volume only, and it indicates the percentage change in volume which is given by:

$$\varepsilon = [(V_i - V_f)/V_i]$$

where: ε = strain

V_i = initial volume

V_f = final volume

Numerical values of strain increase from zero (no compression) and have higher values which approach 1 as the degree of compression increases (i.e., final volume of the product decreases). Since compressive strain is unidirectional (i.e, length and width do not vary), the change in height of the sample can be used instead of change of volume (Emami et al., 1979), such that,

$$\varepsilon = (h_i - h_f)/h_i$$

where: h_i = initial height
h_f = final height

In this text the use of the term strain refers to compressive strain unless specified.

2.4.2.2 Compression Ratio

Compression ratio is defined as the ratio of the initial sample volume prior to compression to its final volume (C.R. = V_i/V_f). The compression ratio thus increases from a value of 1 (no compression) and has higher values as the degree of compression increases (i.e., volume of the final product decreases). Compression ratio indicates the factor by which the volume of the sample has decreased due to compression. Like compressive strain, when the compression is unidirectional (i.e., length and width do not vary), height changes can be used to determine compression ratio such that C.R. = h_i/h_f.

2.4.3 Compression Behavior

In all materials the deformation that occurs is some function of the applied force. For example, linear elastic (Hookean) materials such as an ideal spring (Mohsenin, 1968) have a relation between stress and strain (in general form) which is given by:

$$\sigma = E\varepsilon$$

where σ is the applied stress, ε is the resulting strain, and E is "Young's modulus" (the slope of the straight line relating stress and strain, which defines the stiffness or rigidity of the given material).

In viscoelastic materials, the force deformation characteristics are like elastic materials on short time scales and are like viscous fluids on long time scales (a combined solid-like and liquid-like behavior). Materials of this type are modeled by combinations of springs and dashpots, with the spring element in the mechanical model obeying Hook's law while the dashpot represents a Newtonian liquid. The two basic combinations of these elements, the Kelvin model and Maxwell model, are shown in figure 2-1 (Mohasenin, 1968).

It is generally accepted that for small deformations the rheological properties of solid foods can be described by some sort of complex arrangement of springs and dashpots.

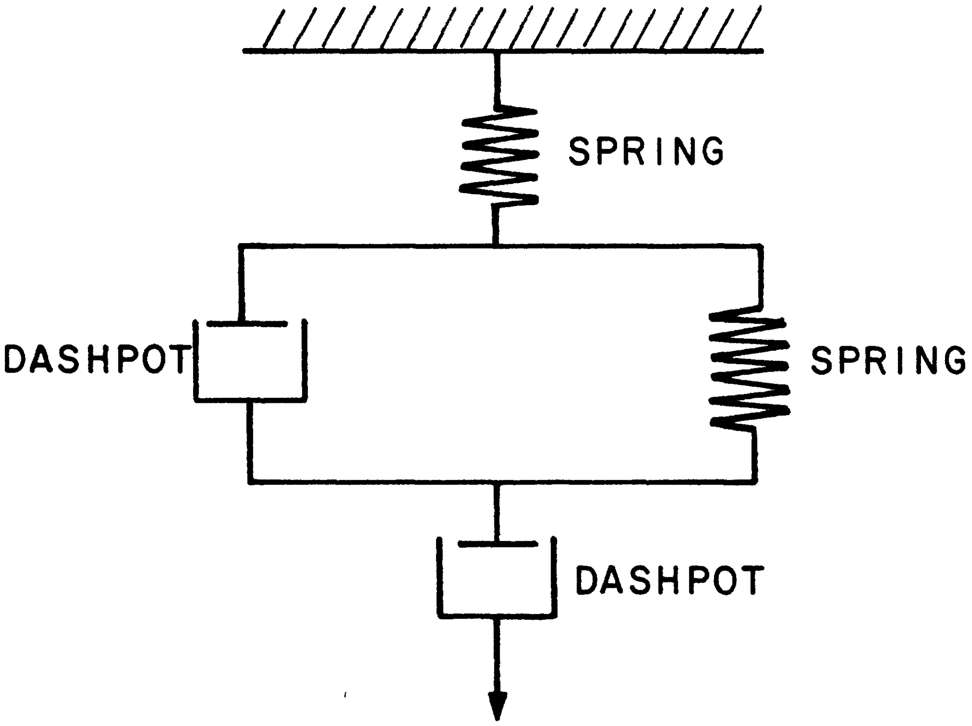


Fig. 2-1 The 4-element model; the basic combination of Maxwell model and Kelvin model.

For large, irreversible deformations in food, deviations from elastic or viscoelastic behavior require the addition of fracture elements to the mechanical models (Drake, 1971; Peleg, 1976).

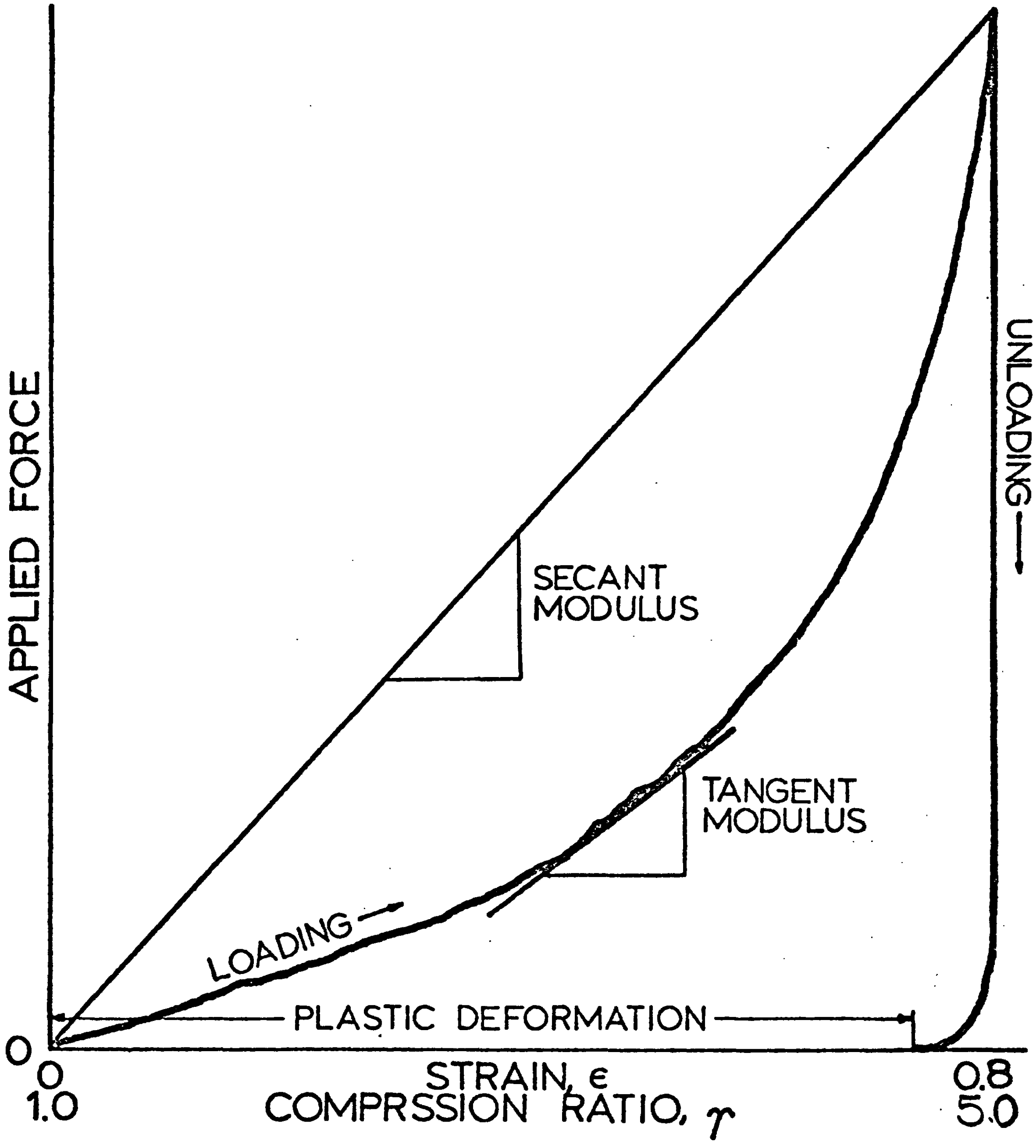
2.4.4 Compression Behavior Analysis

From the force-deformation measurements made with an objective method such as an Instron Universal Testing Machine, numerous quantifiable parameters may be calculated to describe a material's texture. For a uniaxial compression at a fixed crosshead speed (i.e., constant strain rate) of a material with an initial thickness of h_0 and fixed cross-sectional area of A_0, the stress (σ = force/A_0), and the strain ($\varepsilon = (h_0-h)/h_0$), or compression ratio (C.R. = h_0/h) can be obtained from the force-deformation data.

Figure 2-2 shows a typical stress-strain response for freeze-dried, rehumidifed green beans compressed uniaxially with the Instron Universal Testing Machine. The definition of the textural parameters shown in figure 2-2 is given by Mohsenin (1968) as follows:

Secant Modulus:	The ratio of stress to strain at a given point on stress-strain curve (σ/ε). It is an indication of stiffness or rigidity.
Tangent Modulus:	The slope at a given point on the stress-strain curve ($\partial\sigma/\partial\varepsilon$). It is also an indication of stiffness or rigidity of a material.

Fig. 2-2 A typical stress-strain response for freeze-dried, rehumidifed green beans under a uniaxial compression.



Elastic deformation:	Recoverable deformation of a material
Plastic deformation:	Permanent or unrecoverable deformation of a material

2.5 Effect of Moisture and Temperature on Mechanical (Rheological) Properties of Foods (and Other Polymeric Compounds)

Adsorbed water has been found to have pronounced effects on mechanical properties of polymeric materials. However, when the whole spectrum of water contents of the material is considered, the effect of water varies significantly at different levels of adsorption.

At 0% water content, the material is found to be quite rigid, and fragmentation will occur when the material is subjected to stress. At low water adsorption levels, the adsorbate forms strong bonds (e.g., H-bonds, ion-dipole bonds, etc.) to many specific adsorption sites (e.g., hydroxyl groups, carbonyl groups, amino groups, etc.) on the material and does not have its original liquid form (i.e., it is present as "bound" water) (Karel, 1974). At these levels there is no significant effect of water addition on the relative rigidity of the material (G. King, 1950).

At higher water adsorption levels, the relative mobility of polymer chains and the volume they occupy increase (G. King, 1950). It is known that water and other low-molecular-weight compounds (solvents) which can form internal polar bonding or dissolve the polymer (Sakata and Senjue (1975) reduce the local friction coefficient, and increase the mobility of the polymeric segments. Each polymeric segment having adsorbate molecules in its vicinity (as well as other polymeric segments) can be displaced in translatory motion much more easily, thus lowering the effective local viscosity (Ferry, 1961). For the case of most foods, this effect of moisture on relative rigidity is reported for water contents in the range of 5-20 percent. Compression of freeze-dried foods having moisture contents in this range will result in a significant "plastic" deformation (i.e., no recovery after unloading), together with some elastic recovery. The plastic deformation results in elimination or reduction of void volumes which were initially occupied by water. In the compression process, polymeric segments initially separated by these void volumes now have a greater degree of mobility and can be brought closer to one another and be packed without significant breakage or fragmentation. This close packing will result in formation of many polar bonds (e.g., H-bonds) between the polymeric segments (Nissan, 1976) which is responsible for the non-recoverable aspects of the deformation. If the sample is unloaded at the same moisture level used for loading, there will be a time-dependent elastic recovery (i.e., relaxation), which is due to breakage of some of the bonds which were formed during loading (Nissan, 1976). If immediately after compression the sample is further dried (i.e., water is removed), the relative rigidity of the polymeric segments will increase again causing an indefinite deformation so long as the sample is kept dry. On the other hand, if after unloading further water is added (i.e., rehydration), the bonds formed during compression will break at a faster rate, and water migrates back within the polymeric segments, separating them again. The extent of water uptake, which depends on the basic material structure and the strength of the bonds formed during the compression process, will determine the extent to which the sample approaches the original shape and volume.

At high moisture levels the reduction in relative rigidity with increasing moisture levels off (G. King, 1950), apprently since at these levels, liquid water is present in a loose form within the polymeric segments. Compression of the material at high water activities causes collapse or flow which is accompanied by the loss of the original solid structure of the matrix (MacKenzie and Luyet, 1971).

There have been very few studies on compressive rheological properties of foods as a function of moisture content and/or temperature. Kapsalis et al. (1970) reported significant changes in textural properties of freeze-dried, precooked meat when rehumidified to water activities between 0 to 1.0 at room temperature. According to their data, at the range of moisture contents that have been found to be suitable for compression of this product ($\sim$11-12 g H_2O/100 g solids), (Pilsworth, Jr. and Hoge, 1973), the degree of elasticity, secant modulus (σ/ε at 3% compression), toughness (work per unit volume necessary to compress the sample to 25% of its original thickness), and work ratio (ratio of the area under the load curve of the second cycle to the area under the load curve of the first cycle) are much lower than the values of these same rheological properties obtained at other water activities. These results indicate that a greater degree of compresion of precooked beef can be expected at these levels of moisture content. Kapsalis et al. (1970) also indicate that as the B.E.T. monolayer value for water sorption is approached, rigidity (secant modulus), toughness, bioyield strength, work ratio, and degree of elasticity reach a maximum which explains why at very low levels of moisture, foods crumble upon compression.

It should be noted that the effect of water at different adsorption levels is strongly dependent on temperature and basic material structure. Many polymers undergo what is termed a second-order phase transition as their temperature is raised. The change has been described as an internal melting in which the polymer changes from a brittle (glassy) to a rubber-like state, and many of its physical constants alter in value. Presence of moisture (or other low-moelcular-weight solvents) lowers this transition temperature. It is thus possible that increasing concentration of these agents may even cause such structural change when the sample is held at constant temperature (G. King, 1950). At this transition temperature most materials start to flow and lose their original solid structure. Thus, increasing the sample temperature is also able to increase the mobility of the polymeric segments. Sakata and Senjue (1975) reported on thermal compressibility of thiolignin and diaxone lignin in the presence of various low-molecular-weight solvents including moisture. Powdered material containing a predetermined level of the solvent was compressed to form a pellet which was then placed in a flow tester. When the temeprature was elevated at a constant rate of 3°C/min under a constant compressive load of 10 kg/cm^2, they detected three regions of compressive deformation. Noted first was a softening region in which small voids are reduced as the sample gradually deforms under the load. At the softening temperature (T_s), the small voids are eliminated and the pellet becomes a homogenous, transparent material or a single solid phase. At temperatures just above T_s there is little additional compression taking place and no apparent change in the pellet. At some higher temperature (T_f) the flow region is reached and the sample starts to flow and passes through the nozzle which is located in the bottom of the tester.

It should be noted that moisture and temeprature influence the rheological properties of polymers differently depending on the basic material structure. Basic material structure relates to the composition and organization of the components in food. In particular it is concerned with structural polymers (such as cellulose in plants and myofibrils in animal tissue), and those relatively low-molecular-weight compounds that are located between the polymeric segments and have a low glass transition or flow temperatures (such as sugars). For example, sugars seem to be critical in affecting the mechanical properties of plant materials, since they are mostly amorphous after freeze drying and can absorb more water in the amorphous state than the cellulosic materials. At sufficiently high water activities or temperatures, they will soften and flow under a compressive force. If they flow too readily, the cellulosic phase may deform to such an extent that it may lose its ability to retain a reversible strain (MacKenzie and Luyet, 1971). This will cause collapse of the material under the compressive load.

2.6 Methods of Compression and Debulking Dehydrated Foodstuffs

To accomplish compression of foods without shattering and fragmentation of the dried matrix, it is necessary to have the solid structure of the material in a "compressible state." As mentioned earlier (sec. 2.5), depending on the basic material structure, this state can be achieved by manipulation of combinations of moisture content, temperature, and presence of other agents.

In the compressible state the relative rigidity of the polymeric segments within the cellular structure of the food is sufficiently low to allow deformation of the solid matrix without irreversible structural changes under a compressive load.

This state can be achieved by bringing the dry food to a relatively uniform moisture content in the range of 5-20 percent depending on the food product (Hamdy, 1960). Moisture may be transferred either as vapor or sprayed as a liquid mist. It has been shown that the amount of moisture added to the dried product prior to compression significantly affects the rehydrated product (Rahman et al., 1971). For example, as the conditioning moisture increases, the rehydration ratio decreases whereas the resistance to shear becomes greater.

For some products manipulation of temperature alone can be used to achieve a compressible state (Rahman et al., 1970; Do et al., 1975). In some early investigations there have also been attempts to combine the effects of both moisture and temperature to accomplish compression (Donnelly, 1947; U.S.D.A., 1948). Problems associated with thermal treatment are discoloration, burning, and loss of nutritional value of the product.

Agents other than moisture, such as as ethylene, propylene glycol, glycerol, vegetable oil, etc., have been used with some success for compressing different food products (Gooding et al., 1958; Durst, 1967; Ranadive et al., 1974; Pavey, 1975; and others). The major limitation of using these agents is the foreign flavor that they impart to the food.

Once the materials are treated (by humidification, heat, or other agents), they are then placed in a compression cell in a hydraulic press and subjected to uniaxial pressures ranging from 100-5000 psi. The samples are then redried or cooled and packed to yield the compressed product (Rahman et al., 1969).

Freeze-dried foods high in quality, nutritional value, and recovery characteristics can be obtained using the currently practiced methods for producing compressed foods. The method has been described by Rahman et al. (1969) and is shown diagrammatically in figure 2-3.

2.7 Alternative Approaches for Production of Freeze-Dried/Compressed Foods

2.7.1 Limited Freeze Drying

Most developmental work to date for freeze-dried, compressed food has utilized freeze drying to low moisture contents, followed by remoistening of the dry material to attain the desired moisture content for compression. Under certain controlled conditions it is possible to conduct the freeze drying process such that at the "end" of the drying, the material has a uniformly distributed, desired

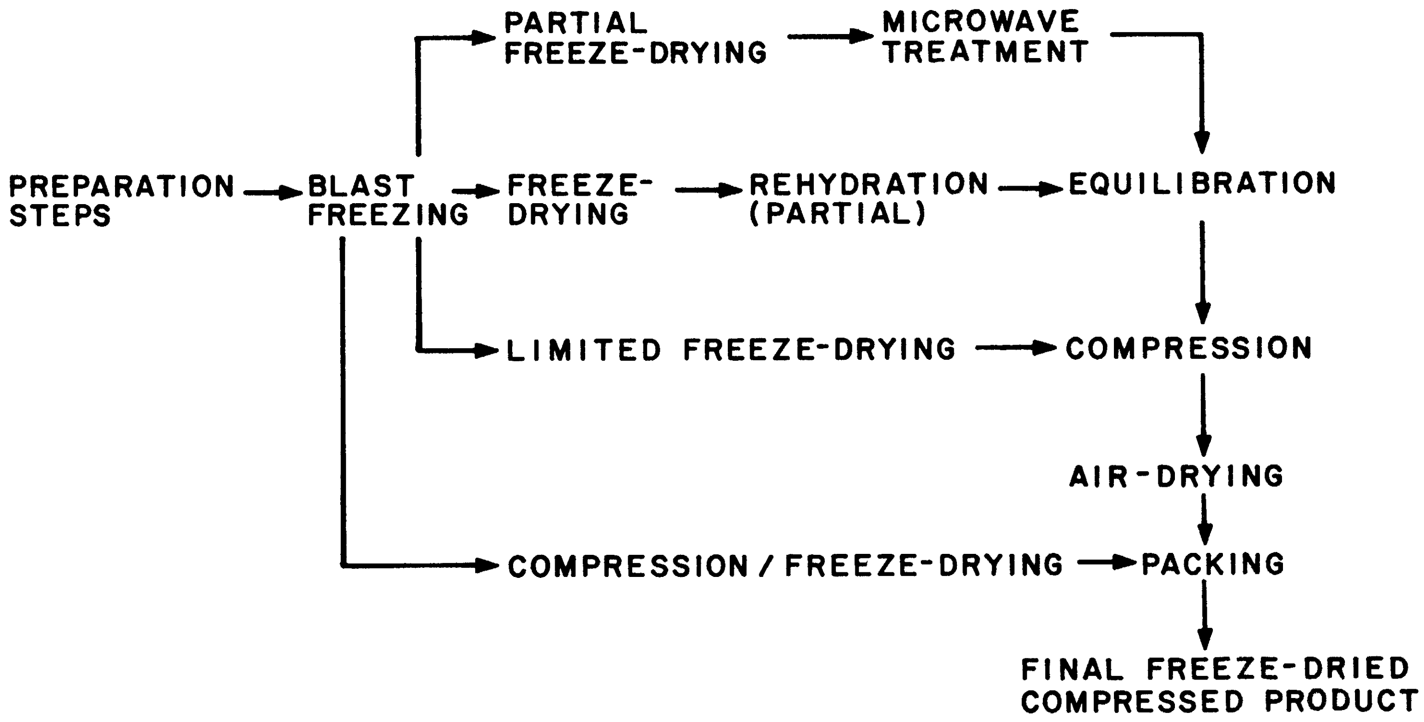


Fig. 2-3. Methods of preparation of freeze-dried/compressed products.

moisture content, thereby avoiding the remoistening step. The term "limited freeze drying" was first applied by MacKenzie to this process (MacKenzie and Luyet, 1969). The principle of the method is to control the freeze drying such that the relative humidity at the surface and within the product does not fall below some predetermined value, which is high enough to ensure that throughout the drying the equilibrium moisture content in all regions of the product will not drop below the desired value. A major limitation in this method is that the sample temperature and the condensor temperature must be controlled within very narrow limits ($\pm$ 0.1°C). Generally, the selection of sample chamber temperatures in the range of -10 to -40°C permits the production of materials containing about 25 to 5 g H_2O/100 g solids, respectively (MacKenzie and Luyet, 1971). To accomplish limited freeze drying through control of environmental relative humidity requires that the temperature difference and water vapor partial pressure differences (driving forces for heat and mass transfer) be less than in ordinary commercial freeze driers, which necessarily leads to longer drying times. King et al. (1975) noted that MacKenzie and Luyet had reported that drying times of 25-100 hours were required for 1 cm cubes of beef and various vegetable products.

King et al. (1975) reported that drying rates for limited freeze drying processes can be accelerated if:

- The heat transfer coefficient from the heat source to the surface of the product is increased.
- The temperature of the heat source is raised during the early part of drying so that the temperature of the product surface rises toward the point where the surface itself experiences the prescribed relative humidity.
- Control of the environmental relative humidity is accomplished through other means than product and condenser temperatures.

They proposed two alternative methods to accomplish a more efficient limited freeze drying process. In one method the heating platen temperature is controlled at the desired final surface temperature, instead of the sample chamber temperature which was originally used by MacKenzie and Luyet (1969, 1971). They demonstrated a 50% reduction in time required for limited freeze drying when compared to a constant platen temperature drying process. A major limitation of this technique is that small fluctuations of the platen temperature will result in a significant change in final moisture content through its influence on the local relative humidity (King et al., 1975).

The second method described by King et al. (1975) is a self-regulating control of the enviornmental relative humidity by using a water-uptake medium which maintains a particular, predetermined relative humidity independent of temperature or amount of moisture taken up. At the end of the drying process, the sample surface and water-uptake medium relative humidity would be the same, provided that food pieces and water-uptake medium are at the same temperature. Jones and King (1975) described a system with alternating layers of food and hydrating salt desiccant. A circulating low-pressure gas is used to transfer heat and mass between the food and desiccant. Since the latent heat of absorption for most desiccants is about equal to the latent heat of sublimation for the same amount of water, the gas will be cooled as it passes through the food layer and then will be dried and reheated as it passes through the desiccant layer.

In the process of limited freeze drying when the food is removed from the freeze drier, it is ready for immediate compression and subsequent final drying to low moistures as shown diagrammatically in figure 2-3.

2.7.2 Microwave Technique

In this approach compression of food products could also be achieved without the need for the initial overdrying and humidification steps which are required by the conventional processing techniques. This technique is based on stopping the freeze drying process at a time when the amount of ice remaining in the food is approximately 20%-25% by weight of the total solids. The condition of uniformly distributed moisture which is required for compression is obtained by vaporizing the remaining ice by a microwave treatment. Haralampu (1977) reported that partial drying of peas to 20%-25% moisture could be achieved, reproducibly, at a platen temperature of 162°F (72°C) and a drying time of 12.5 hours. Microwave treatments at low power levels, but high energy inputs,were sufficient to vaporize the remaining ice and redistribute it relatively uniformly throughout the sample so that compression could be achieved without damage to the solid matrix. He noted that the microwave processed product compared well with conventionally compressed product, with regard to rehydration ratio, fines production and volume recovery. Besides eliminating the overdrying and humidification steps, this process also eliminates the slowest and most costly period of the freeze drying process--the desorption phase when all the ice has been sublimated. Once the microwave treated samples are compressed, they are further dried to produce the dried/compressed product (figure 2-3). Problems associated with this technique include reproducibility of achieving the desired final moisture content for the partial freeze drying, and discoloration, and burning that can occur during the microwave treatment when higher power levels are used.

2.7.3 Simultaneous Freeze Drying and Compression

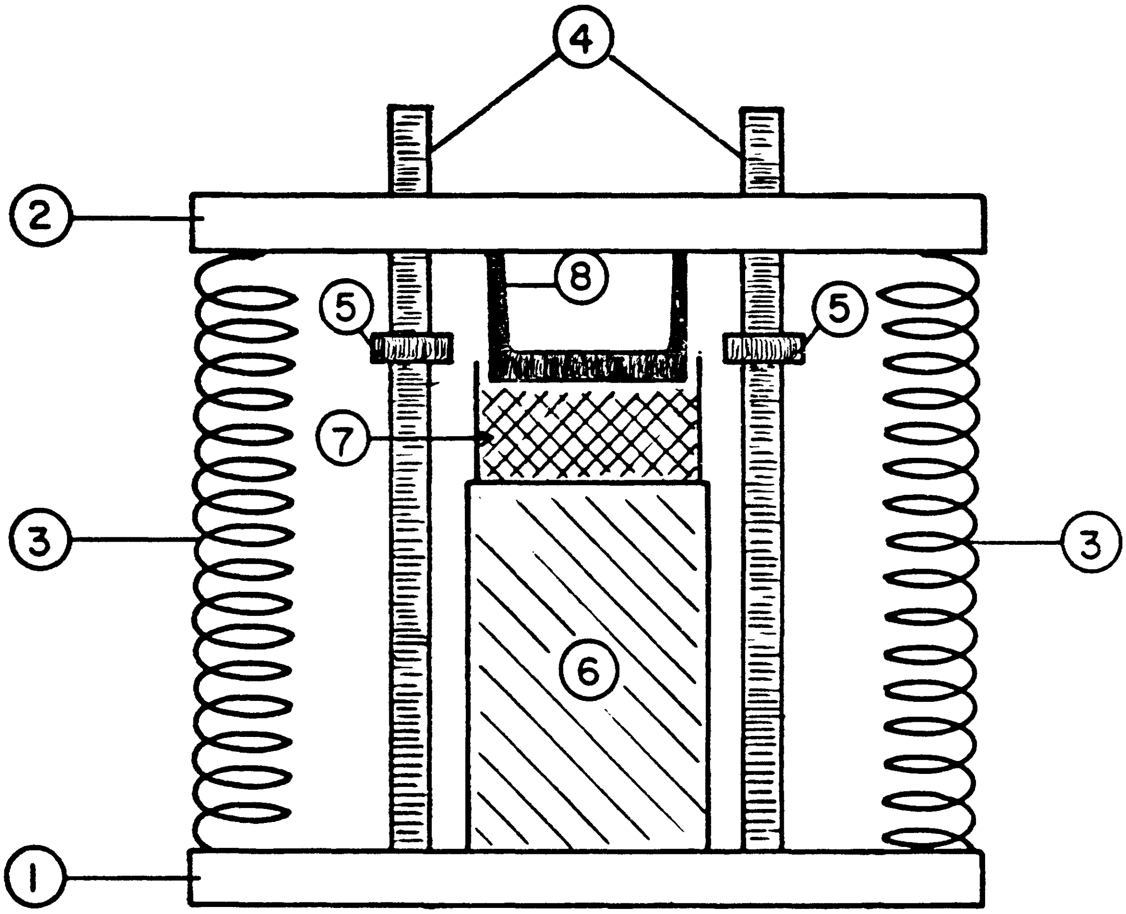
The phenomenon of simultaneous freeze dehydration and compression was first reported by Konigsbacher (1974). In this approach the food material is subjected to compressive force during the freeze drying process such that drying and compression of the food material take place simultaneously. This avoids many processing steps such as overdrying, rehumidification, redrying and other treatments that are required in the other techniques used to produce freeze-dried/compressed foods (figure 2-3). A wider demonstration of the feasibility of this technique has been made by Emami (1976). He noted that his results indicated that during freeze drying there exists enough moisture in the diffusion zone at the ice-dry layer interface to make it suitable for compression. Application of force thus results in a gradual compression of the interface as the drying of the food material proceeds to low moisture contents.

A spring-driven device was used to provide the required compressive pressures in the range of 5-185 psi (figure 2-4). The results found by Emami et al. (1979) are summarized in the following sections (2.7.3.1 to 2.7.3.6).

2.7.3.1 Compression Behavior

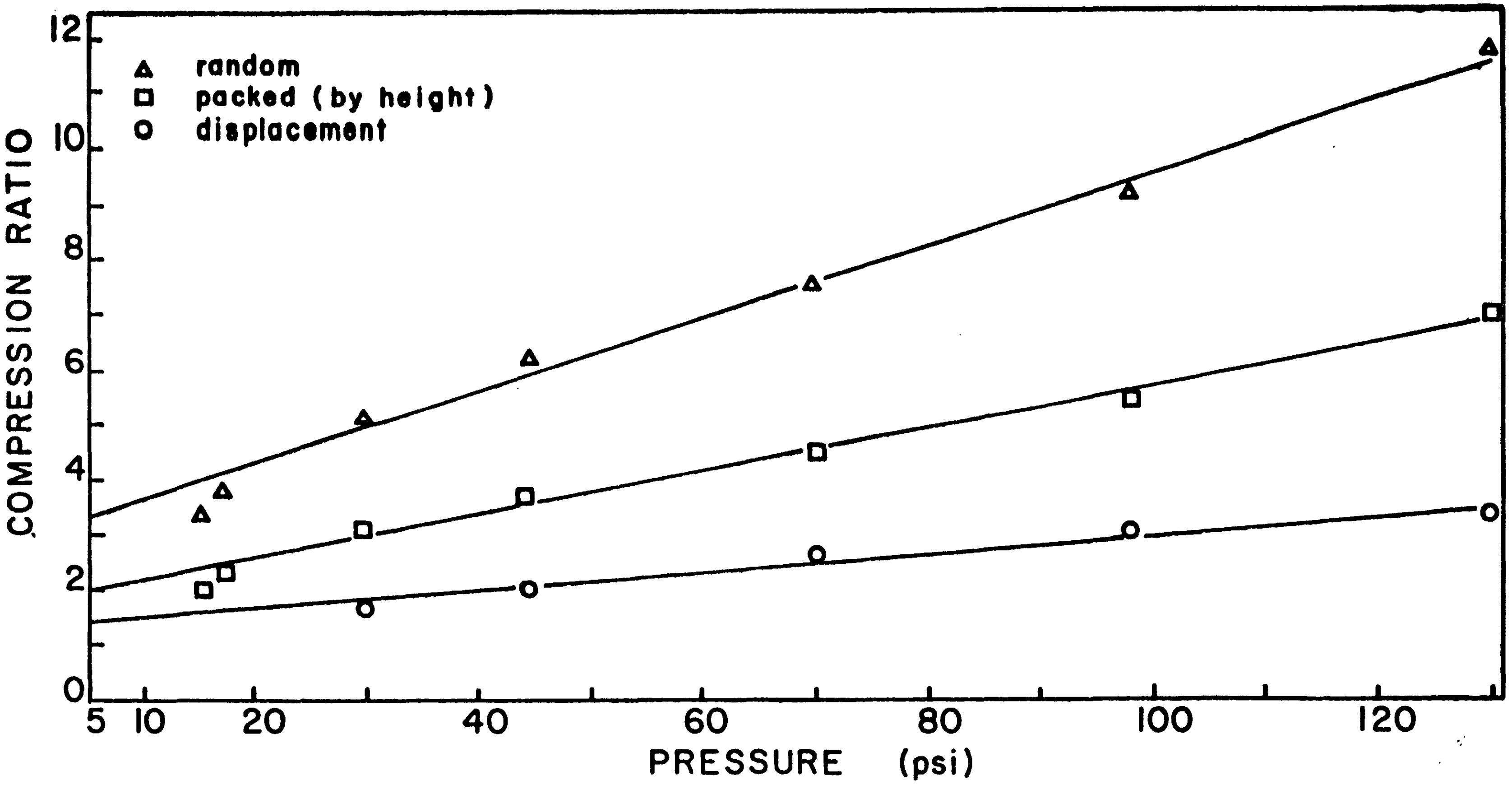
For a variety of food materials tested at fixed sample thicknesses, the compression ratio was linearly related to compressive pressure over the range of pressures tested. However, the actual compression ratio obtained at a given force was dependent on the particular food item. Figure 2-5 illustrates the relationship of compression ratio and compressive pressure obtained for green beans. Theoretical considerations would indicate that for a reversible compression (i.e., without destroying the solid structure), the compression ratio will reach a maximum value at some compressive pressure so that no increase in compression ratio

Fig. 2-4. Schematic of spring compression system (loaded).



1	Base Plate
2	Top Plate
3	Matched Set of Springs (Extension Type)
4	Threaded Guide Rods
5	Nuts for Raising Top Plate
6	Sample Cell Stand
7	Sample Holder
8	Compression Plate

Fig. 2-5. Effect of compressive pressure on compression ratios of green beans for spring system.



occurs with further increase in compressive pressure. It is thought that this maximum compression ratio, which reflects the "true" volume change within the given sample piece and not the voids between individual pieces, is directly related to the initial water content of the food material. As noted earlier, this is measured by the "displacement" compression ratio.

2.7.3.2 Effect of Sample Thickness

When the initial bed thickness was varied for green beans, a plot of compression ratio versus sample thickness resulted in two linear, parallel lines corresponding to the two fixed compressive pressures tested. As shown in figure 2-6, compression ratio decreased linearly with increasing sample thickness for each given compressive pressure. Since the slopes are equal, this decrease in compression ratio is apparently independent of compressive pressure, and apparently is due to geometry effects for a bed of green beans when packed in the holder. In packing cylindrical bodies, as the number of layers are increased, the ratio of end layer void volume to the total void volume will not stay constant. This results in increased packing density of the sample, since as the number of layers increases, the void volumes between the pieces are a smaller percentage of the total volume. A similar view is obtained by noting that when packing cylindrical bodies such as green beans in a rigid holder, a lower packing density of material at

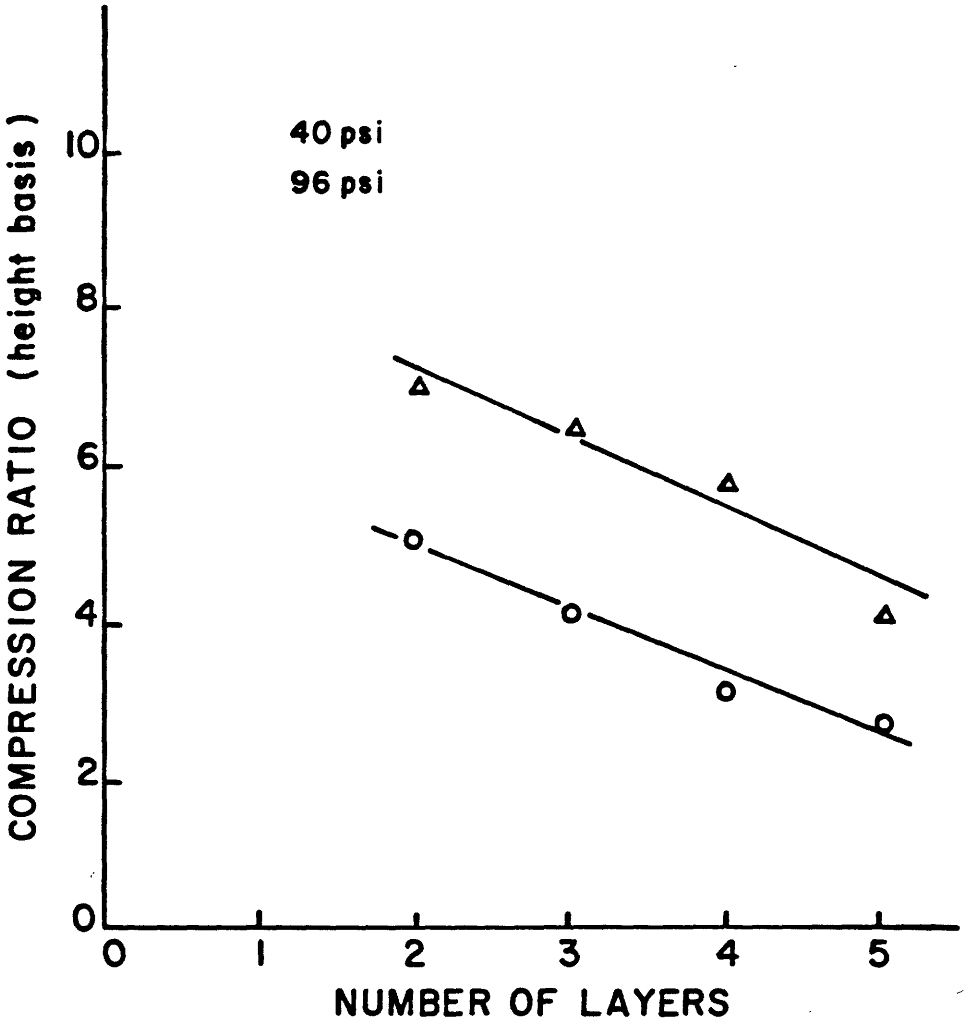


Fig. 2-6. Dependence of compression ratio on sample thickness for green beans.

the walls of the sample holder, or in this case the top and bottom plates, will result. As "internal" layers are added, the effective initial packing density of the entire bed increases.

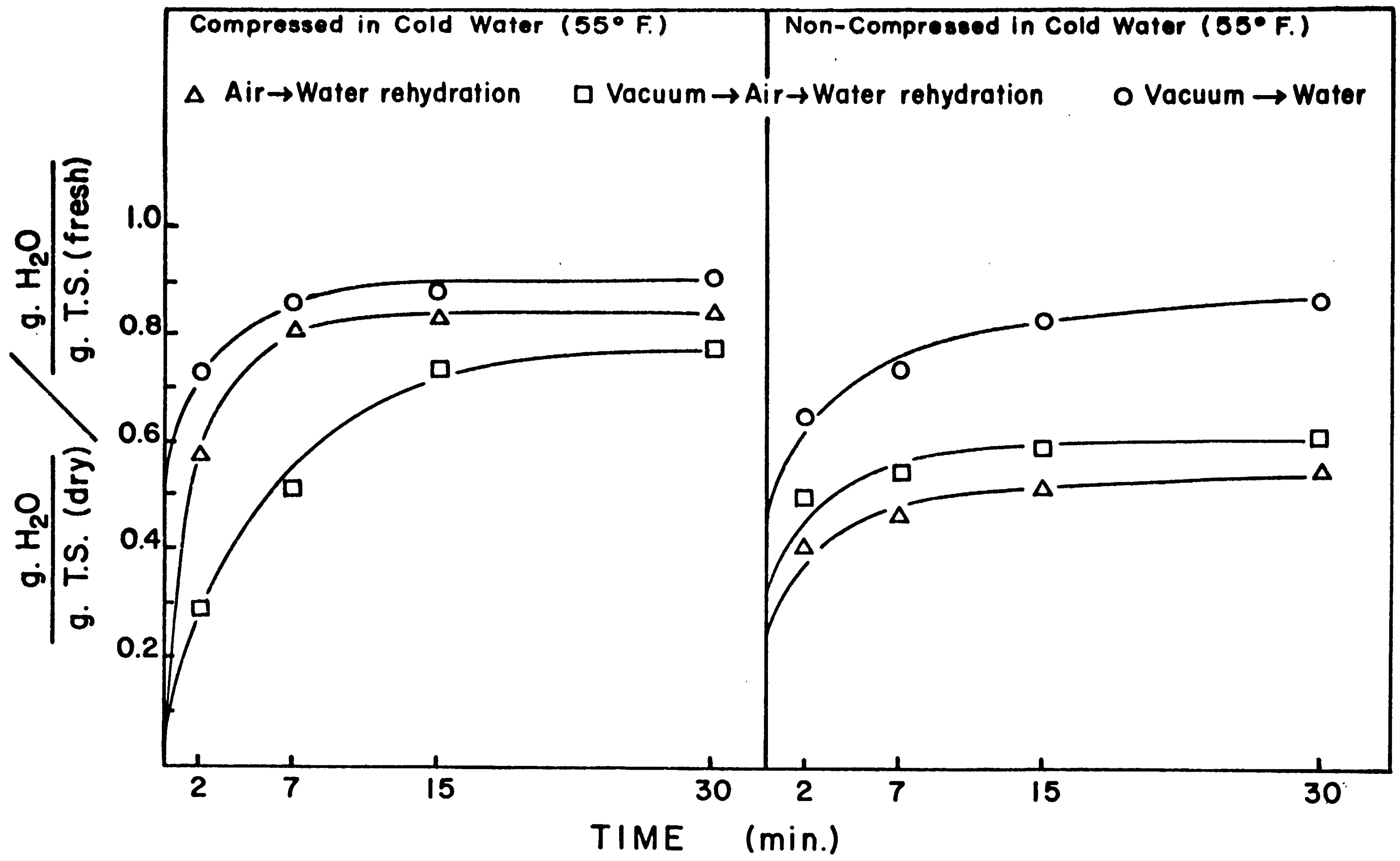
2.7.3.3 Final Moisture Contents

Emami (1976) showed that the final moisture contents of compressed samples were always similar to those of non-compressed controls, freeze-dried at the same time. In most cases the final moisture contents were in the range of 1.5-5 percent, which indicated that compression during freeze drying did not prevent dehydration to low moisture levels. It was further noted that final moisture contents did not seem to depend on the compression ratio.

2.7.3.4 Rehydration Behavior

Emami (1976) also investigated the rehydration of compressed samples and showed it was quick, in most cases reaching equilibrium within 15 minutes. Samples generally reattained their precompression volume after rehydration. It was shown that when just immersed in water, freeze-dried, non-compressed vegetables generally rehydrated to a lesser extent than compressed samples. However, evacuation of the freeze-dried samples prior to addition of water showed little effect for compressed samples, whereas the non-compressed samples showed a marked increase in rehydration (figure 2-7). Vacuum rehydration increased the extent of

Fig. 2-7. Effect of mode of water addition on rehydration of freeze-dried green beans.



rehydration of non-compressed vegetables to that of compressed samples. These results indicate that the lower uptake of water by non-compressed vegetables was due to air entrapped in the cells and that some structural changes which are caused by compression affect either removal of the entrapped air in the cells or water uptake by the cells resulting in improved rehydration behavior.

It was also noted that for vegetables, the extent of rehydration was not affected by the degree of compression (figure 2-8), whereas beef cubes showed a slight trend toward reduced rehydration with increasing compression ratio (figure 2-9).

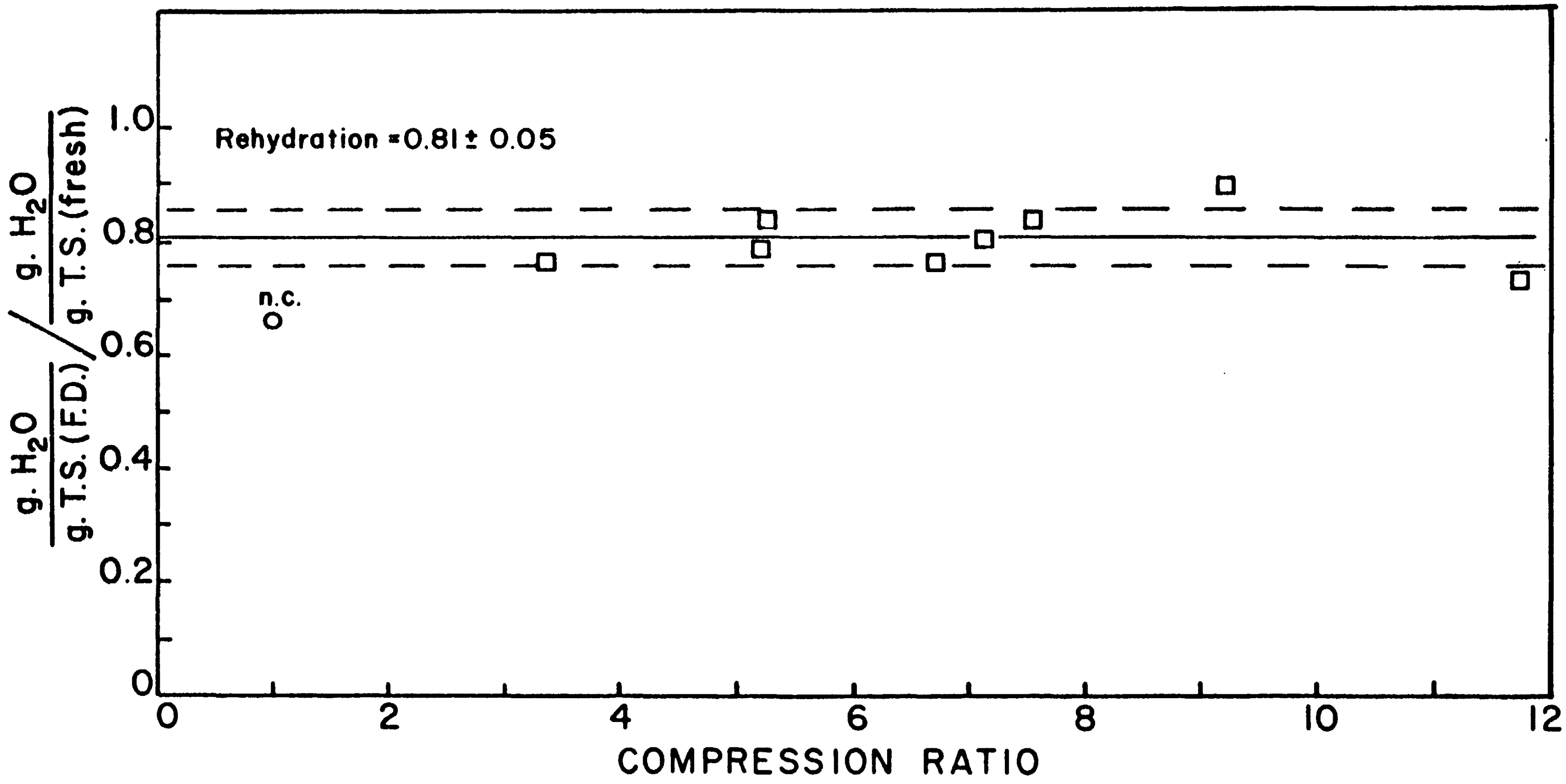
2.7.3.5 Organoleptic Evaluation

Organoleptic evaluations of cooked, non-compressed, compressed, and fresh or frozen samples indicated that compressed samples were always rated equal to or better than the respective non-compressed samples. Fresh samples were usually rated higher than either of the freeze-dried products, though in most cases the differences were not significant at a 1% level.

2.8 Moisture-Temperature Gradients in the Dry Layer during Freeze-Drying; Their Effects on Mechanical Properties of the Dry Layer during Freeze Drying

State of water during freeze drying has already been discussed (section 2.3). The idealized distribution of moisture during sublimation is that shown in figure 2-10a (Karel, 1975).

Fig. 2-8. Effect of the degree of compression on rehydration of freeze-dried, compressed breen beans.



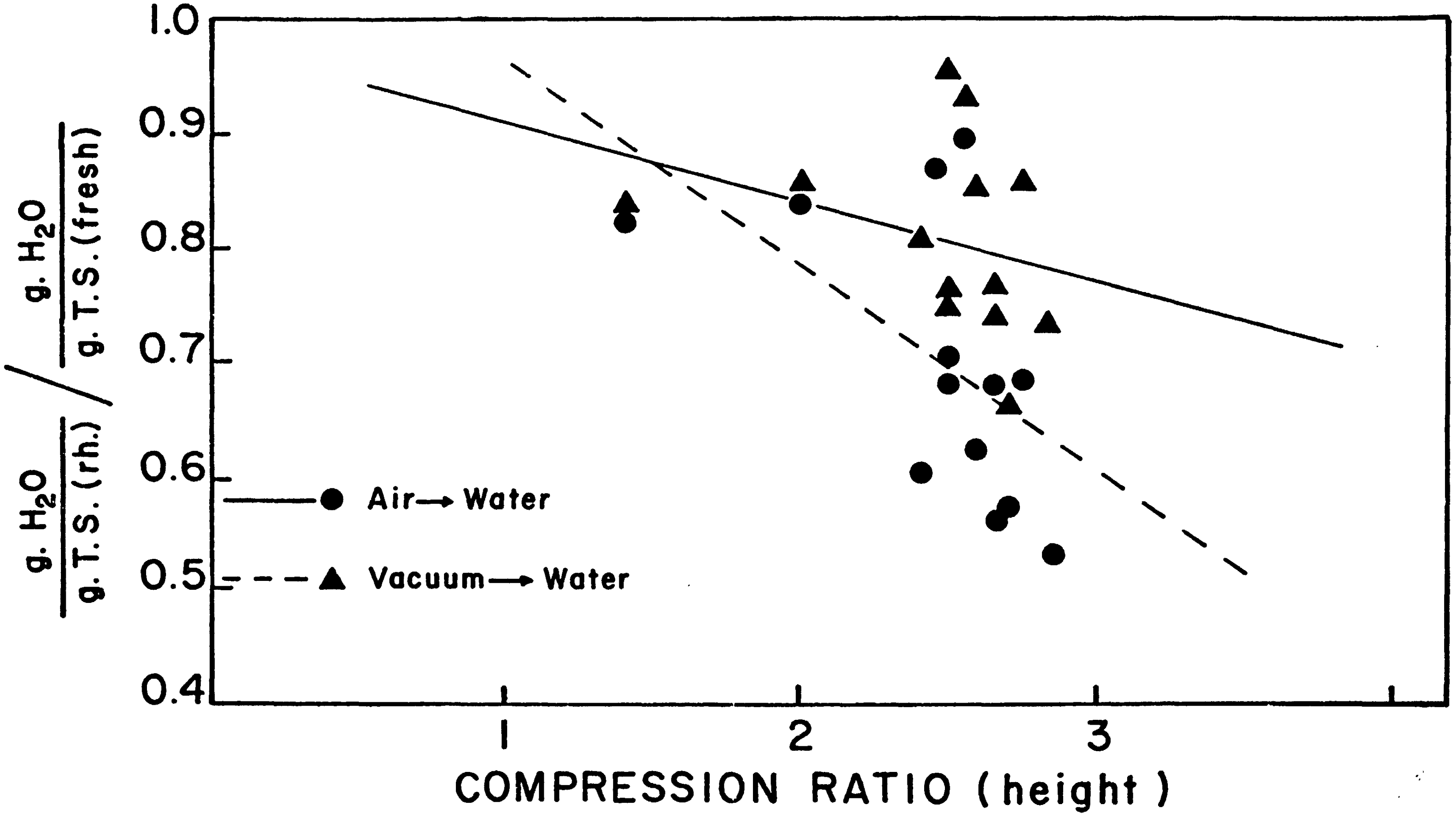
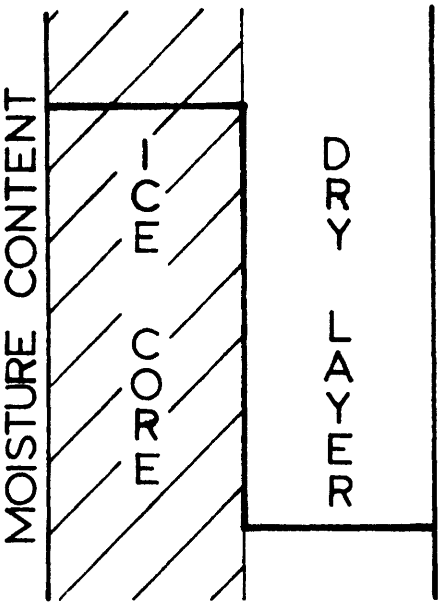


Fig. 2-9. Effect of the degree of compression on rehydration of freeze-dried, compressed beef cubes.

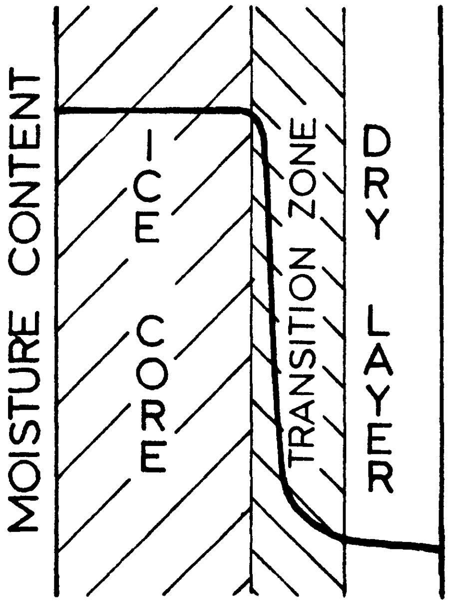
in which there is an interface at which the moisture content drops from the initial level (m_0) in the frozen layer to the final moisture content of the dry layer (m_f). The final moisture content (m_f) is determined by the equilibrium of the sample solids with water at the partial vapor pressure of water in the space surrounding the dry layer. However, evidence given by many authors indicates that this is actually not the case, and the moisture profiles are represented more accurately by figure 2-10b, which shows the existence of some gradient in the "dry layer" (Karel, 1975).

Temperature histories during freeze drying of different materials have been reported by many investigators (Kessler, 1962; Sandall et al., 1967; King, 1971; Aguillera and Flink, 1974; and others). Their data indicate a very thin transition between a frozen zone of uniform low temperature, and a dry zone with a nearly linear gradient of temperature between the ice front and the outer surface.

Gentzler and Schmidt (1973) reported a linear relationship between log of the local moisture content at any position in the non-ice portion (referred to by the authors as the bound water at that position) and the temperature difference between that position in the dry layer and the temperature of the frozen region during freeze drying of skim milk. Their relationship was based on higher desorption temperatures for bound water as compared with the sublimation temperature for frozen water. Aguilera and Flink (1974)



(a) IDEALIZED GRADIENT



(b) PROBABLE GRADIENT

Fig. 2-10. Models of moisture gradients present during freeze drying (from Karel, 1975).

showed data of Oetjen (1973) for coffee grounds gave similar behavior. This behavior is shown in figure 2-11, for both the skim milk and granulated coffee studies.

From figure 2-11 it can be seen that if the temperature of the frozen region remains constant during the freeze drying process, then at any instant in time, the local temperature and moisture content of any location in the dry layer zone are directly related, and thus for each given temperature in the dry layer there will be a unique value of moisture content at that location whose value can be determined from figure 2-11 (for skim milk and granulated coffee).

Aguilera and Flink (1974) report that for freeze drying with constant surface temperature, a "rotation" of the temperature gradient occurs about the constant surface temperature point, resulting in decreasing temperature gradient as drying proceeds. Since the moisture content remaining after the passage of the ice front is directly related to the difference between local temperature and the ice front temperature (as described above), higher moisture contents can be expected at a given distance from the ice front as drying proceeds. This is in accordance with descriptions of Bralsford (1967) and Brajnikov et al. (1969) of a soft wet broadening zone between the ice zone and the dried layer, since intermediate levels of moisture build up continuously against the front as drying proceeds.

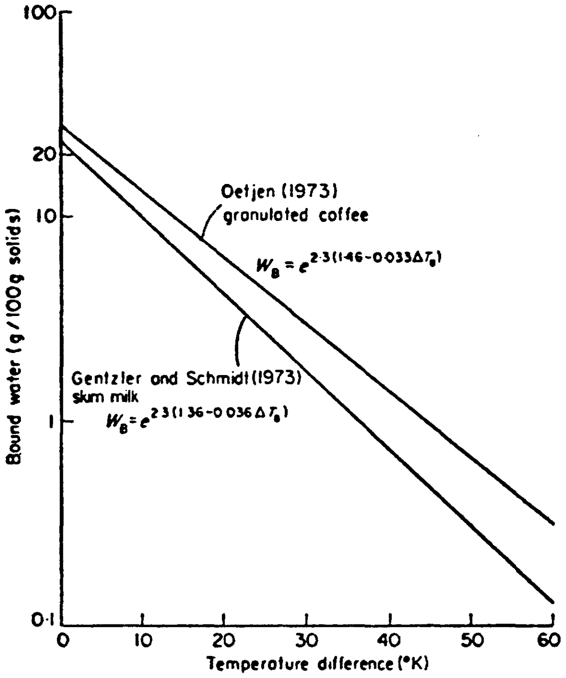


Fig. 2-11

Local moisture contents in the dry layer during freeze-drying as a function of difference between local temperature and ice-front temperature (from Aguillera and Flink, 1974)

The effects of moisture and temperature on mechanical properties of foods and other polymeric compounds were described previously (section 2.5). By combining these effects with moisture and temperature gradients and their relationship in the dry layer and at the ice front, it can be expected that at any instant in time, during freeze drying, each location will have different and fixed mechanical properties which is a direct function of the moisture content and temperature at that location. Furthermore, since moisture content and temperature of each location in the dry layer will change as the drying proceeds, the mechanical properties of that location will constantly change with time during the freeze drying process. It can be expected that at any instant in time, if a constant compressive load is applied to the food during freeze drying, each location in the dry layer will undergo a different degree of compression. These considerations which have not been investigated previously are an important aspect to the compression of foods during freeze drying and are investigated in this thesis.

3. EXPERIMENTAL

The experimental procedures have been divided into two major parts. The first part presents procedures for experiments involving compresion during freeze drying, while the second part presents the materials and methods used in evaluation and characterization of the compressive rheological properties of product as a function of moisture and temperature (referred to as "Instron studies").

3.1 Equipment for Compression during Freeze Drying

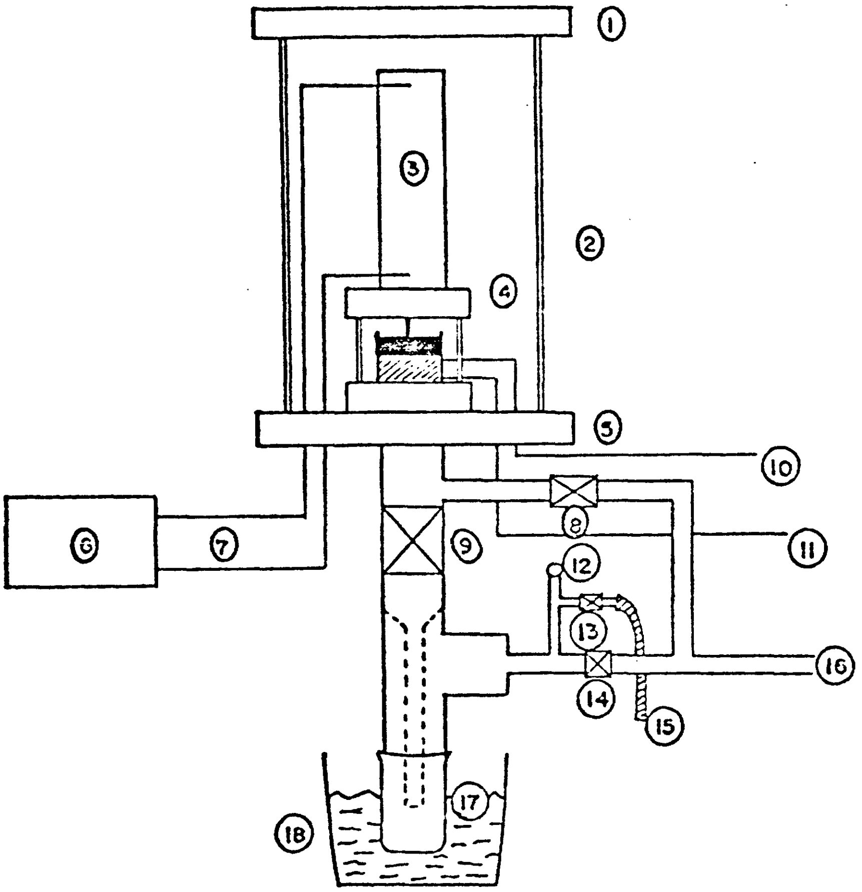
3.1.1 Hydraulic Compression System

The hydraulic compression system was designed for complete insertion within the freeze dryer chamber. A condensor system was developed which allowed collection of the sublimed ice so that drying rates could be determined, since direct weighing was not possible due to the large mass of the hydraulic cylinder and cell support assembly. A schematic of the hydraulic compression system is given in figure 3-1.

The hydraulic compression system operates by means of an air driven pump (SC Pump Model 10-500-3), which develops hydraulic pressures from 0-2500 psi from laboratory air up to 45 psi.

Control of the air pressure regulator determines the fluid force (up to 5000 lbs) applied on the piston of the

Fig. 3-1. Schematic of the hydraulic compression system.



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[1]
1. Chamber Top (plexiglass)
2. Chamber
3. Hydraulic Cylinder
4. Sample Holder
5. Chamber Base
6. Hydraulic Pump
7. Hydraulic Fluid Lines
8. Valve (bypass)
9. Vacuum Isolation Valve
10. Thermocouple Wires
11. Heater Power
12. Vacuum Gauge
13. Valve
14. Valve
15. Drierite Tube
16. To Vacuum Pump
17. Condensate Collector
18. Chilled Alcohol Bath

hydraulic cylinder (Hennels Co., Model HH-144-N-2X1). Thus while the maximum compression pressure in the hydraulic cylinder is fixed, the ultimate compression pressure available at the sample will be related to the sample holder surface area. (If required, this system can be easily converted to higher forces or more sensitivity at lower forces by changes in air pumps or cylinders.) To distribute the hydraulic force, the cylinder is fixed to a top plate-base frame and the cylinder rod acts on the "compression plate" of the sample holder.

3.1.2 Vapor Trapping System

Figure 3-1 also shows the schematic for the vapor trapping system which permits determination of drying rates during compression/freeze drying. The vapors sublimated from the sample are condensed in a stainless steel vessel immersed in a chilled alcohol bath. The stainless steel vessel is removed from the trapping system by isolating the chamber (which remains under vacuum) from the condenser and bleeding in dry air (close valve 9, 14; open valve, 8, 13). A transfer of condenser vessels can be achieved in about 30 seconds with very little increase in chamber pressure. A material balance showed in almost all cases that the collected condensate is approximately equal to the material sublimated.

3.1.3 Sample Cell Holder

Sample holders have been constructed of mesh or perforated metal sheets having different perforation sizes, and percentage open areas to facilitate vapor transport through the walls as well as the top and bottom surfaces. A schematic of the sample cell holder is shown in figure 3-2. Parts A and C are termed top and bottom compression plates, respectively. The fine wire thermocouples connected to these plates are used to both monitor temperature of the plates and as inputs to the heater controllers which are controlling the amount of heat input to the sample being freeze-dried/ compressed. In most experiments the percentage open area of these plates was chosen to be 63% (maximum available) to maximize vapor transport through the top and bottom surfaces.

The top and bottom compression plates rest on coarse metal gratings which provide sturctural support for the system when it is under hydraulic force, while not hindering moisture transport.

The cell wall (Part B) is constructed either from the same perforated metal sheets as the compression plates (to obtain moisture transport from the sides) or from solid material (no opening) in order to obtain a more uniform drying with moisture loss from the top and bottom surfaces only (i.e., closer to the U.R.I.F. model).

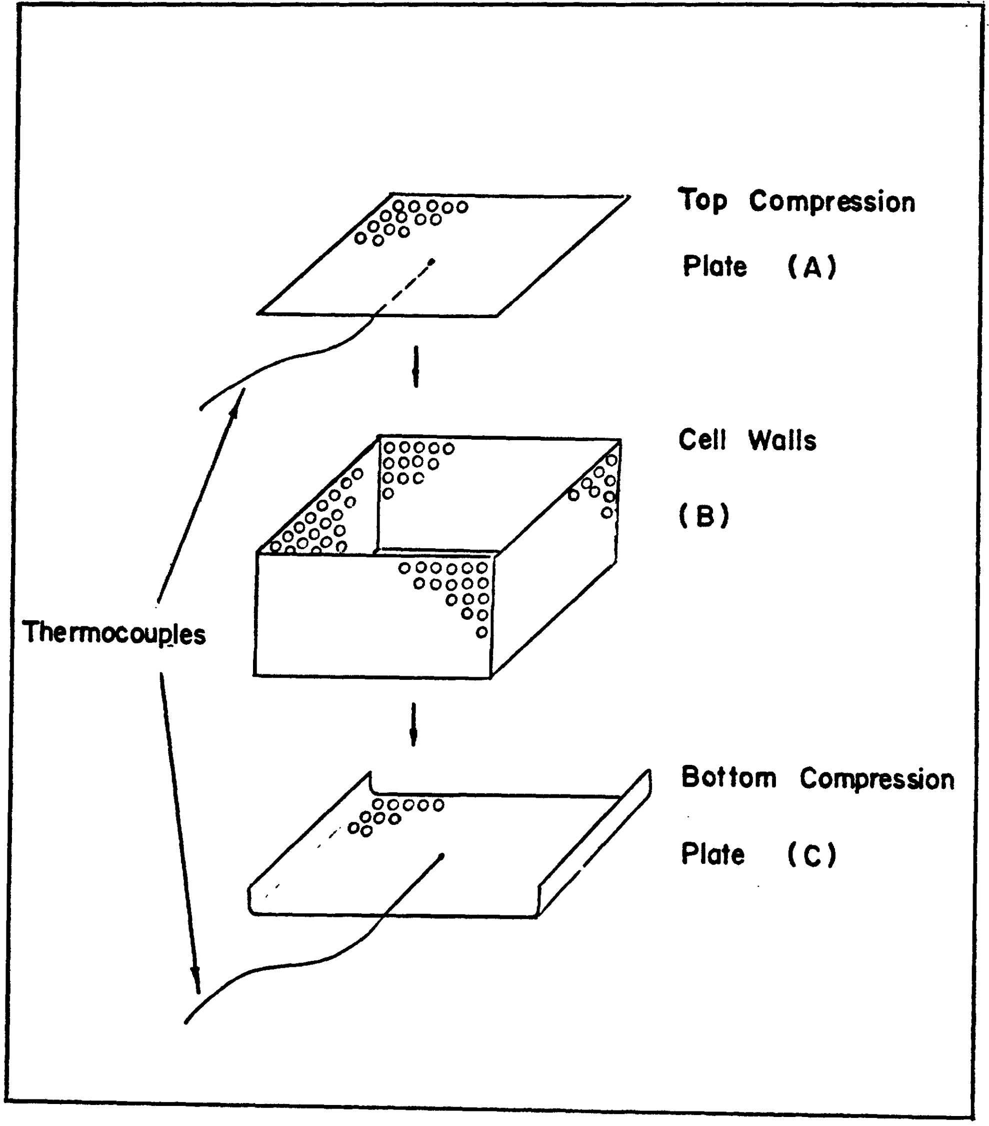


Fig. 3-2. Schematic of the sample cell holder for compression during freeze drying experiments.

The sample cell holders were constructed in three different sizes (7.5 x 7.5 cm^2, 10.5 x 10.5 cm^2, and 15 x 15 cm^2) in order to evaluate the effect of size on compression and freeze drying of foods.

In operation, the bottom compression plate (C) rests on a metal grating which is stationary while the top compression plate (A) moves downwards inside the cell walls (B) as the sample is being compressed. The location of the top compression plate (and thus the sample thickness) is monitored continuously by measuring the voltage drop across a linear variable resistor which is connected between the hydraulic cylinder support and top compression plate.

When controlled heat input is desired, silicon rubber heaters are placed above the top and below the bottom metal support gratings mentioned earlier. Electrical energy to the heaters is controlled by thermocouples located either on the heaters or at chosen locations in the sample. Thus, for this system, the heat transfer is both by conduction (through the metal gratings and compression plates), and radiation (through open spaces of the metal gratings and perforated metal sheets).

3.1.4 Sample Loading

Cut, frozen green beans purchased at a local supermarket were hand sorted to obtain regular cylindrical pieces of adequate dimensions. The green bean pieces were stacked in the sample holder in three layers like a pile of logs so that the compression force was only in the radial direction.

3.1.5 Program for Compression during Freeze Drying Experiments

The experimental program is designed in such a way that the effects of parameters relevant to compression and drying behavior of the product could be investigated independently. The experiments conducted in this phase of study are listed in table 3-1.

As can be seen from table 3-1, parameters of interest are compressive pressure, heat input, and size and porosity of the sample cell.

3.1.5.1 Compressive Pressure or Loading

Compressive pressure denoted in psi indicates the amount of pressure load delivered by the hydraulic compressor on the sample, this pressure being held constant at a given value throughout the experiment. In order to evaluate the dependency of the degree of compression on sample loading or the applied compressive pressure, different levels of applied compressive pressure were used in different sets of experiments as shown in table 3-1. Furthermore, since compression of the dry layer during freeze drying is expected to result in improved heat transfer

Table 3-1 Program for Compression during Freeze Drying Experiments

Set #	Compressive pressure (psi)[a]	Top and bottom compression plate temperature (C°)[a]	Porosity of the cellholder walls (% Open Area)	Porosity of the top and bottom compression plates (% Open Area)	Cell holder size (cm x cm)	No. of bed layers
1	0 (non-compressed), 20, 30, 40, 50, 60	no external heat input	∿60%	∿35%	7.5 x 7.5	3
2	0 (non-compressed), 30, 50, 60, 70	∿-4°C	63%	63%	7.5 x 7.5	3
3	30, 50, 60	∿+4°C	63%	63%	7.5 x 7.5	3
5	20, 40, 60, 70	0°C	63%	63%	7.5 x 7.5	3
5	40	10°C	63%	63%	7.5 x 7.5	3
5	40	0, 10, 20°C	0%	63%	7.5 x 7.5	3
5	20	0, 10°C	0%	63%	7.5 x 7.5	3
5	40	0, 10°C	63%	63%	10.5 x 10.5	3
5	40	0, 10°C	0%	63%	10.5 x 10.5	3

[a] Each compressive pressure and/or temperature denotes one experiment (e.g., total of 6 experiments in set #1, etc.)

(due to increased thermal conductivity of the dried layer and reduced pathway length for heat transfer), this effect must also be considered.

3.1.5.2 Heater Controls/Energy Input

Heater controls relate to the amount of energy being supplied into the system in order to accelerate the process. The temperatures indicated in table 3-1 correspond to set points for the controlling thermocouples located at the inner side of the top and bottom compression plates (in touch with sample surfaces) as shown in figure 3-2. Evaluation of the influence of this process variable provides information on maximum or optimum heating plate temperatures and/or surface temperatures to accelerate drying rates, achieve high degrees of compression and yet not exceed the limits for a given set of mass transfer and pressure load conditions at which irreversible structural changes begin to occur.

3.1.5.3 Sample Holder Size and Porosity

Size refers to the length and width of the sample holder used in this study. These dimensions were chosen such that the surface areas have a ratio of 1:2:4 (i.e., 7.5 x 7.5 cm^2, 10.5 x 10.5 cm^2, and 15 x 15 cm^2), which also means that the weight loading in these holders has the same ratio.

Porosity is the percentage open area of the sample holder cell walls (figure 3-2). As mentioned earlier the percentage open area of the top and bottom compression plates was fixed at 63% which is the maximum porosity available for metal sheet of sufficient strength. Porosity of the cell walls were varied as shown in table 3-1.

These variables (size and porosity) relate to potential mass transport behavior, both in percentage open surface area and relative diffusion distance in the x-y vs. z planes. To overcome the effect of compression on the potential for vapor buildup in the dried/compressed layer and reduce the effect of compression plate on the drying surface, sample holders and compression plates of special design are required so that the material can be subjected to surface forces without extensively impeding the flow of vapor from the drying surface.

3.1.6 Terminology for Compression during Freeze-Drying Experiments

Henceforth, parameters for a given experiment will be denoted in the following manner:

dimension, % open area, °C, psi

For example, 7.5, 0%, 10°C, 40 psi would mean an experimental setup where the size of the sample holder is 7.5 x 7.5 cm^2, the side walls are solid (0% open area), the controlling thermocouples on the top and bottom compression plates are held at 10°C, and the amount of compressive pressure is 40 psi held constant throughout the experiment.

3.2 Materials and Methods Used in Evaluation of the Rheological and Mechanical Properties of Greenbeans as a Function of Moisture and Temperature (Instron Studies)

3.2.1 Freeze Drying

Cut, frozen green beans purchased at a local supermarket were hand sorted to obtain regular cylindrical pieces of adequate dimensions as before. A conventional laboratory freeze drier (Virtus, Uni-Trap Model 10-K) was used to freeze dry unilayer batches of green beans to low moistures at room temperature and a chamber pressure of below 50 millitorr. After freeze drying, the green beans were kept under vacuum in a desiccator over Drierite before rehumidification.

3.2.2 Rehumidification

Seven saturated salt solutions of known water activity (a_w) (table 3-2) were placed in the bottom of seven desiccators. The freeze-dried green beans were then placed above the salt solutions in mesh containers. The desiccators were evacuated to hasten the equilibration process. The desiccators remained covered at room temperature (∿20°C) for about one week.

Table 3-2 Relative humidites used for rehumidification of freeze-dried green beans for studies of mechanical properties

Salt	%RH
Drierite (not in solution)	0
MgCl	33
$NaBr_2$	58
CuCl	66
NaCl	75
$(NH_4)_2SO_4$	81
KNO_3	93

Source: Labuza et al, 1976

3.2.3 Compression with the Instron Universal Testing Machine

A compression cell made of plexiglas (figure 3-3) with dimensions of 2 cm x 3 cm was used for compression tests. A compression block, also made from plexiglas, was constructed to exactly fit in the compression cell. The cell was designed so that three green bean pieces having approximate dimensions of 2 cm x 3 cm would make one layer (figure 3-3). Three layers of green beans were used for each test (i.e., total of 9 pieces).

The rehumidified samples of green beans at different relative humidities were packed in the compression cell as shown in figure 3-3 and quickly adjusted to the desired test temperatures (-25°C, -10°C, +15°C, and +24°C) before compression. Eight to ten samples (9 beans each) were compressed at each specific combination of moisture content (RH) and temperature.

An Instron Universal Testing Machine (Model 1122), equipped with a 500 kg tensile-compressive load cell, was used for compression tests. The applied force was recorded as a function of the crosshead position with full scale readings of 10, 20, 50, 100, 200, and 500 kg.

The three layers of green beans were compressed uniaxially to a final thickness of 3 mm at a crosshead speed of 20 mm/min. To evaluate the effect of crosshead speed, in another set of experiments, freeze-dried green

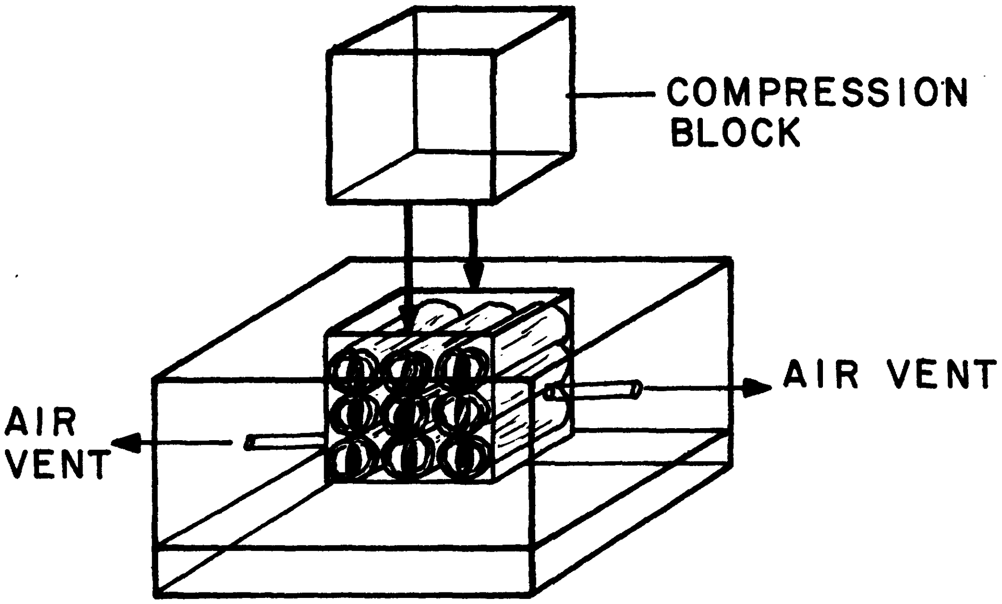


Fig. 3-3. Schematic of the sample cell holder for compression tests using the Instron Universal Testing Machine.

beans rehumidifed at different relative humidites were compressed at room temperature (∿24°C) at a crosshead speed of 50 mm/min. Once compressed, the stress-strain behavior during unloading was immediately measured (i.e., no dwell time). The crosshead speed in the reverse direction was the same as the downward direction.

After the compression, the sample was dried in a vacuum oven at 65°C (150°F) for 24 hours for determination of the moisture content.

4. RESULTS AND DISCUSSION

Analysis of the experimental results have also been divided into different parts to follow the organization of the experimental procedures. The major categories include: results of experiments involved in compression of green beans during freeze drying, evaluation of the compressive properties of this product as a function of moisture content and temperature (Instron studies), and finally correlation of the behavior of compression during freeze drying to the mechanical properties of this product.

4.1 Compression during Freeze Drying Results

Table 4-1 shows the overall results obtained in different sets of experiments conducted at different values of the parameters mentioned in section 3.1.5.

4.1.1 Dependence of the Degree of Compression on Sample Loading; Compressive Pressure-Strain Relationship

The dependence of the degree of compression expressed as strain ($\varepsilon = \Delta L/L$) on the amount of compressive pressure applied (compressive pressure-strain relation) during freeze drying is illustrated in figure 4-1 for different sets of experiments. It can be seen that at low levels of compressive pressure (i.e., less than 10 psi), very little or no compression can be expected to occur for green beans, and a minimal amount of compressive load is required to

Table 4-1 Results of Compression/Freeze-Drying Experiments Conducted with Green Beans at Different Freeze-Drying Conditions

	Compression pressure (psi)	Top and bottom plate temperature (°C)	Porosity (% Open Area)[a]	Cell holder size (cm x cm)	Initial moisture content (g H_2O/ 100 g solids)	Time of drying (hours)[b]	Strain $\varepsilon=\Delta L/L^c$	Compression Ratio $\gamma=L_0/L^c$
Set #1								
Run #1	20	no external heat input	60%, 35%	7.5 x 7.5	1000	28	0.57	2.3
Run #2	30	"	60%, 35%	7.5 x 7.5	970	28	0.63	2.7
Run #3	40	"	60%, 35%	7.5 x 7.5	1020	29	0.66	2.9
Run #4	50	"	60%, 35%	7.5 x 7.5	1040	28	0.68	3.2
Run #5	60	"	60%, 35%	7.5 x 7.5	920	27	0.69	3.3
Set #2								
Run #2	30	∿-4°C	63%, 63%	7.5 x 7.5	1000	21	0.67	3.0
Run #1	50	∿-4°C	63%, 63%	7.5 x 7.5	1040	25	0.74	3.8
Run #5	60	∿-4°C	63%, 63%	7.5 x 7.5	980	12	0.77	4.4
Run #3	70	∿-4°C	63%, 63%	7.5 x 7.5	980	28	0.79	4.9
Set #3								
Run #1	30	∿+4°C	63%, 63%	7.5 x 7.5	950	28	0.70	3.4
Run #2	50	∿+4°C	63%, 63%	7.5 x 7.5	1030	24	0.75	4.1
Run #3	60	∿+4°C	63%, 63%	7.5 x 7.5	1000	14	0.78	4.6
Set #5								
Run #1	20	0°C	63%, 63%	7.5 x 7.5	750	27	0.54	2.2
Run #2	40	0°C	63%, 63%	7.5 x 7.5	850	21	0.72	3.6
Run #3	60	0°C	63%, 63%	7.5 x 7.5	750	17-18	0.75	4.1
Run #4	70	0°C	63%, 63%	7.5 x 7.5	890	15	0.79	4.9

Table 4-1 (continued)

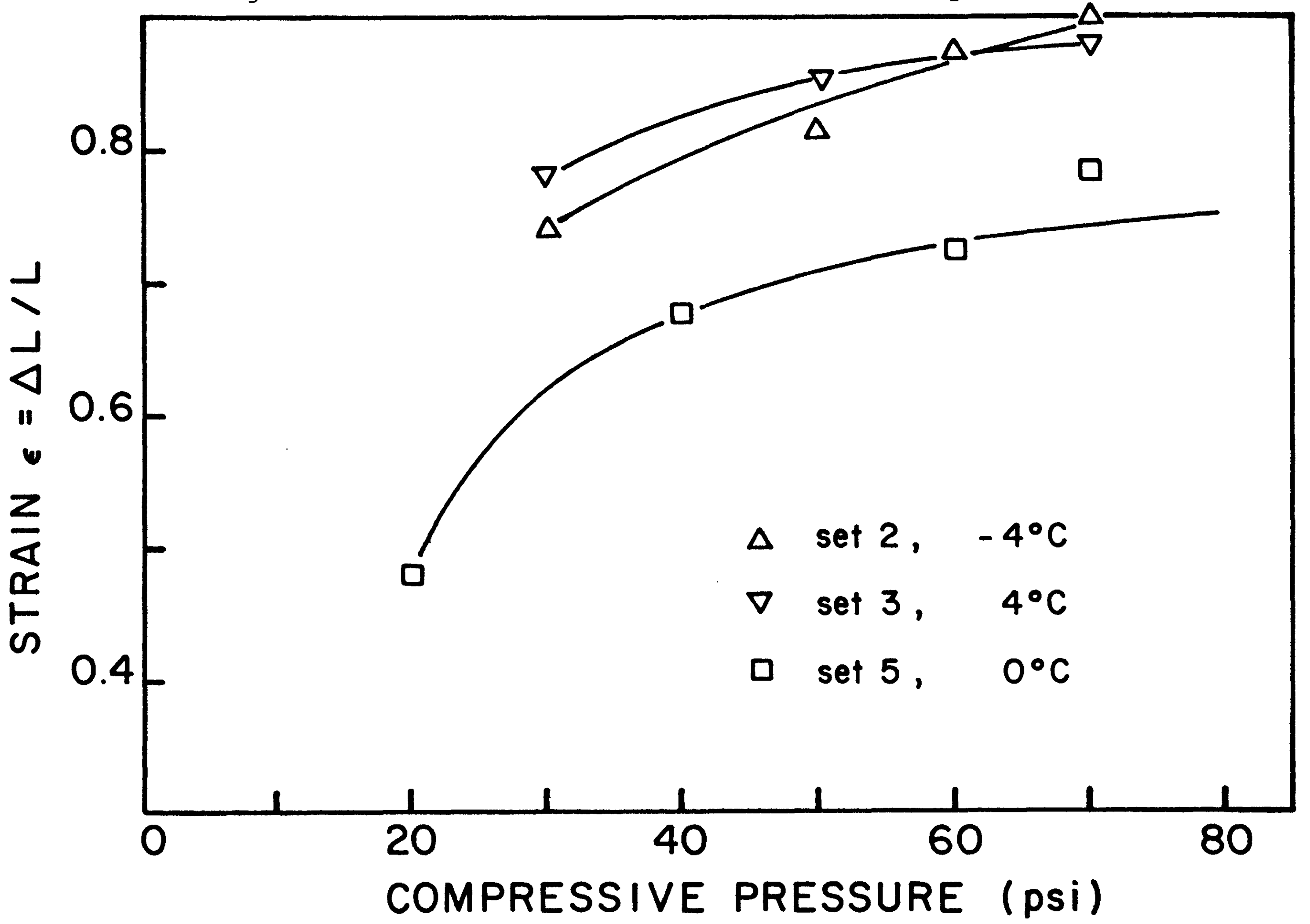
	Compression pressure (psi)	Top and bottom plate temperature (°C)	Porosity (% Open Area)[a]	Cell holder size (cm x cm)	Initial moisture content (g H_2O/ 100 g solids)	Time of drying (hours)[b]	Strain $\varepsilon=\Delta L/L^c$	Compression Ratio $\gamma=L_0/L^c$
Set # 5 (cont.)								
Run #7	40	10°C	63%, 63%	7.5 x 7.5	790	18	0.73	3.7
Run #9	40	10°C	63%, 63%	10.5 x 10.5	820	21	0.71	3.4
Run #10	40	10°C	0%, 63%	7.5 x 7.5	800	19	0.73	3.0
Run #11	20	10°C	0%, 63%	7.5 x 7.5	790	24	0.56	2.3
Run #12	40	10°C	0%, 63%	10.5 x 10.5	1090	18	0.71	3.4
Run #13	20	10°C	0%, 63%	7.5 x 7.5	920	24	0.55	2.2
Run #14	40	10°C	0%, 63%	10.5 x 10.5	900, 760	20	0.72	3.5
Run #15	40	0°C	0%, 63%	7.5 x 7.5	915	24	0.72	3.5
Run #17	40	0°C	0%, 63%	10.5 x 10.5	960	22	0.71	3.4
Run #18	40	20°C	0%, 63%	7.5 x 7.5	930	13-15	0.73	3.7
Run #19	20	0°C	0%, 63%	7.5 x 7.5	930	27-28	0.56	2.3

[a] First value indicates porosity of the cell walls and second indicates that of the top and bottom compression plates.

[b] In set #5 the time of drying is given as the time of drying to reach final moisture content of 5%.

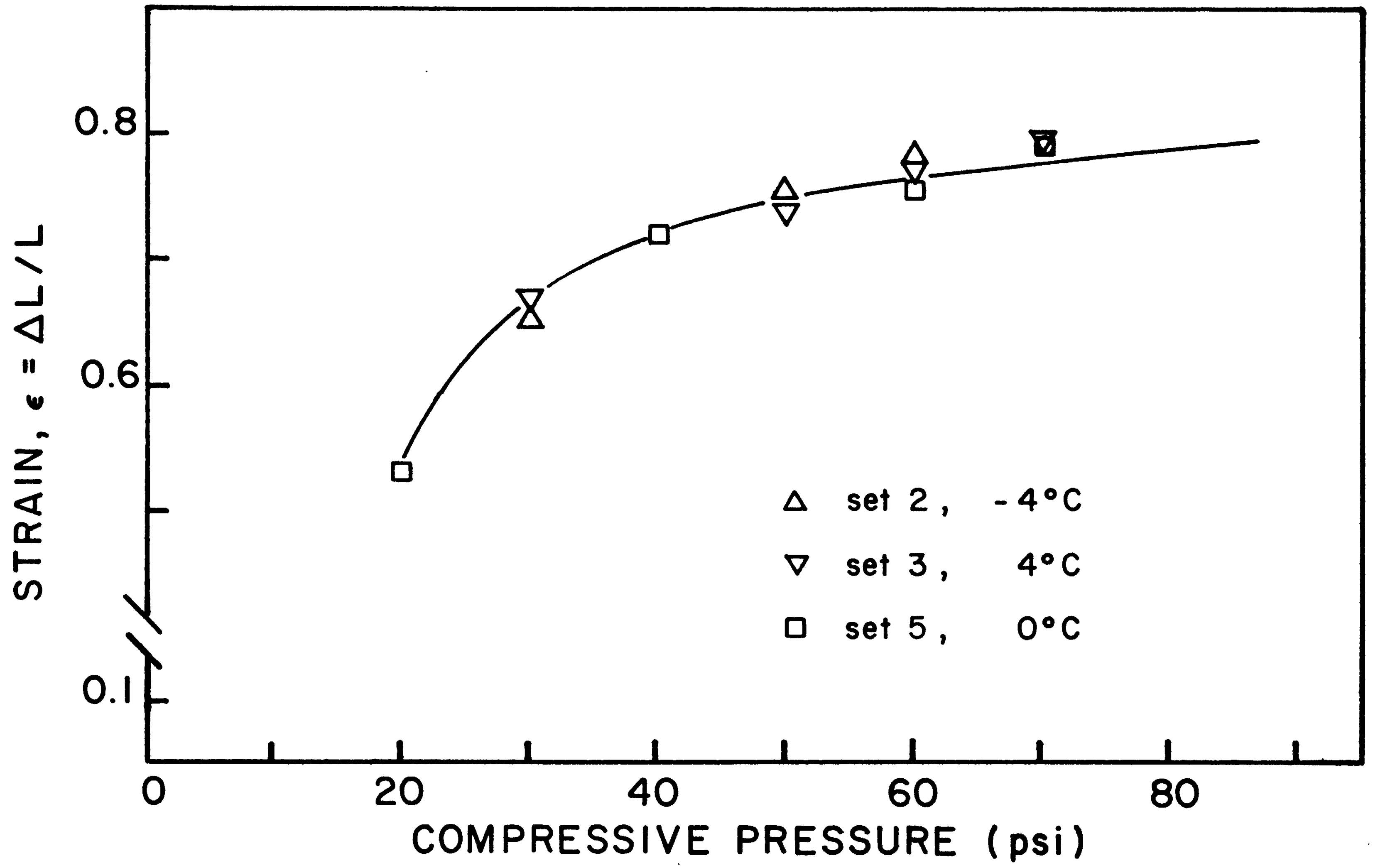
[c] Corrected for initial moisture content.

Fig. 4-1. Effect of compressive pressure on degree of compression (strain) of green beans at different levels of heat input.



initiate compression. This behavior is similar to the results reported in the literature obtained with a spring compression system (Emami, et al., 1979). Another point to be noted in figure 4-1 is that even though the temperature set points are different for each experimental set, the degrees of compression obtained for lower temperature settings (e.g., set #2 vs. set #5) are somewhat higher. The only apparent variable parameter in the experiments shown in figure 4-1 was temperature and since it can be seen that the variation of temperature in different sets does not have any particular trend, the results in figure 4-1 were considered somewhat surprising. However, a closer examination of the _initial moisture content_ of the green beans used in these experiments indicated that a wide variation existed among different sets. Figure 4-2 shows the same experimental results after correcting moisture levels to an initial moisture content of about 900 g H_2O/100 g solids. It can be seen that in figure 4-2 all the data points superimpose at corresponding levels of the applied pressures. The fact that the initial moisture content can affect the final degree of compression at a given set of process conditions, relates to the void volumes initially occupied by water. For a given sample it can be expected that the higher the initial moisture content (and consequently a greater volume occupied initially by water), the higher the degree of compression at a given applied compressive pressure, since compression

Fig. 4-2. Effect of compressive pressure on strain (corrected for initial moisture content) of green beans at different levels of heat input.



of these void volumes does not require any force. This indicates that initial moisture content has to be taken into account when compressing during freeze drying.

Figure 4-3 shows the degree of compression expressed as compression ratio versus compressive pressure. The data points in this figure have been corrected for the initial moisture content. It can be noted that the compression ratio increases linearly with compressive pressure over the range of pressures tested. This confirms earlier results obtained for green beans and other products with the spring compression system (Emami et al., 1979); when food is under an applied compressive pressure during freeze drying, it will undergo a deformation X until the resistive force, -KX, is equal to the applied compressive force. At this point the sample will undergo no further compression, which is similar to the behavior of a spring with a constant modulus under stress.

Figure 4-4 shows the compression behavior as a function of time during freeze drying at varying levels of applied pressure. It can be seen that the reduction in volume has a uniform rate and is essentially linear with time at a given applied compressive pressure; however, as can be expected, the rate of compression increases as the applied compressive pressure is increased. By the end of the process, compression levels off and reaches a final value.

Fig. 4-3. Effect of compressive pressure on compression ratio (corrected for initial moisture content) of green beans at different levels of heat input.

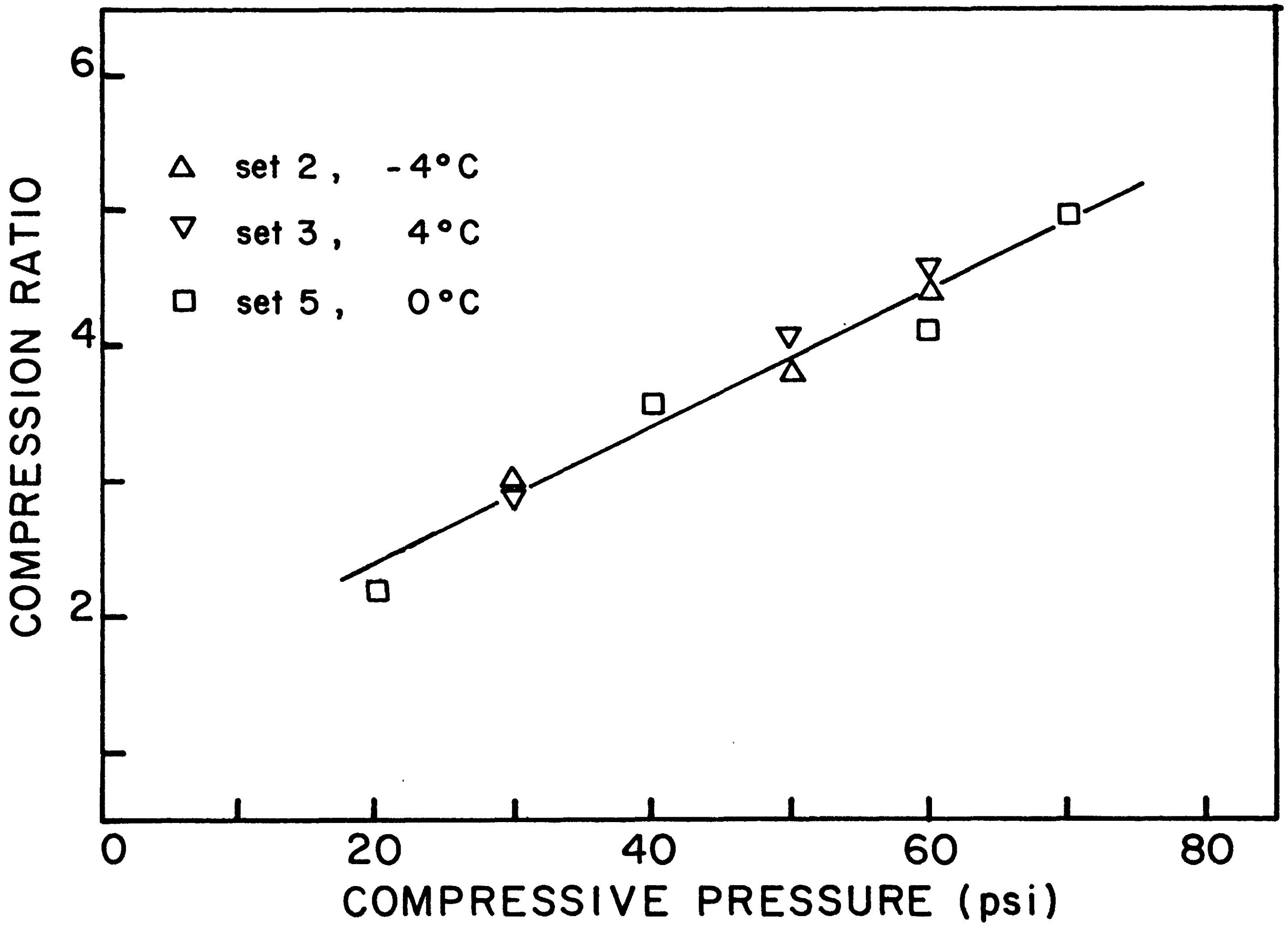
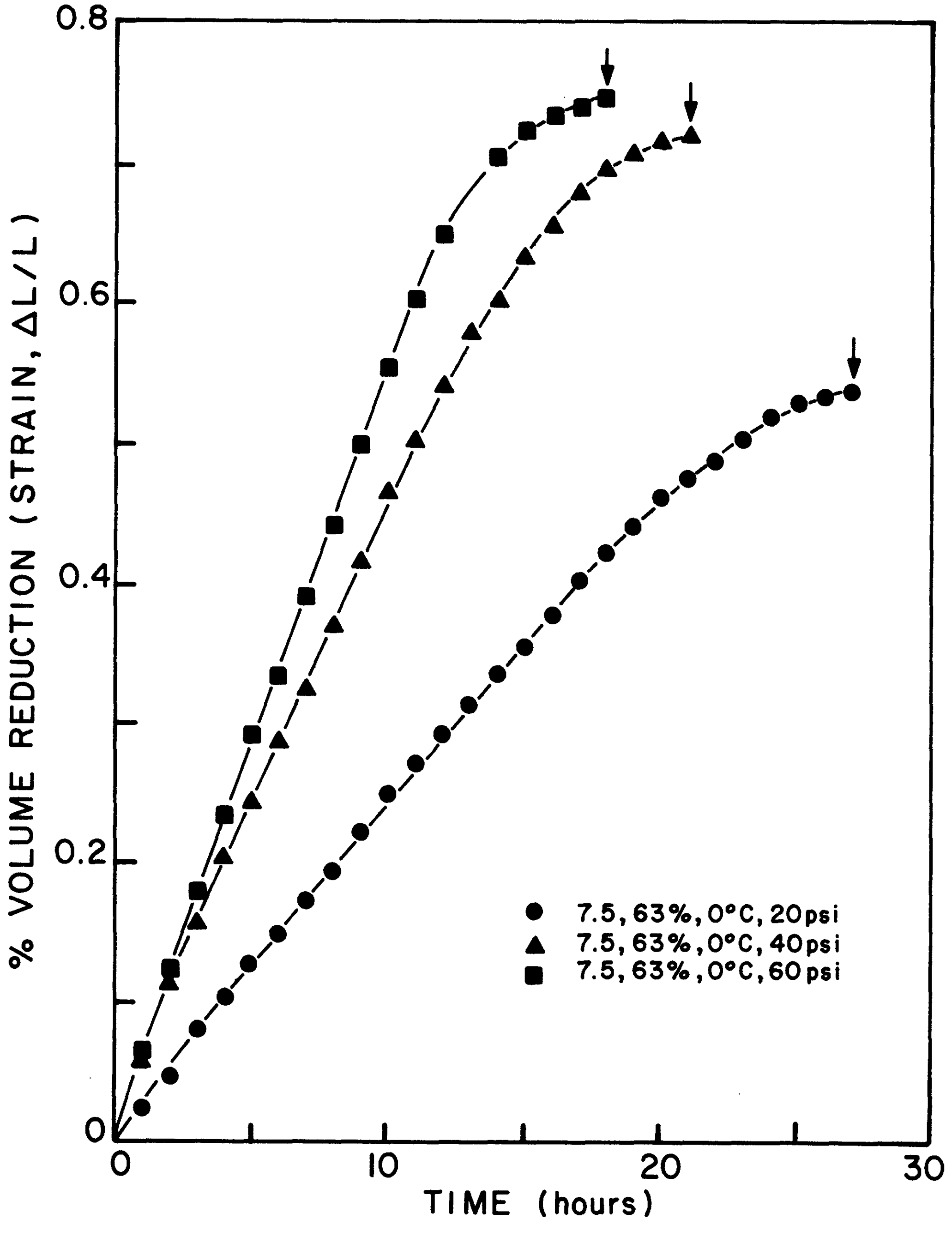


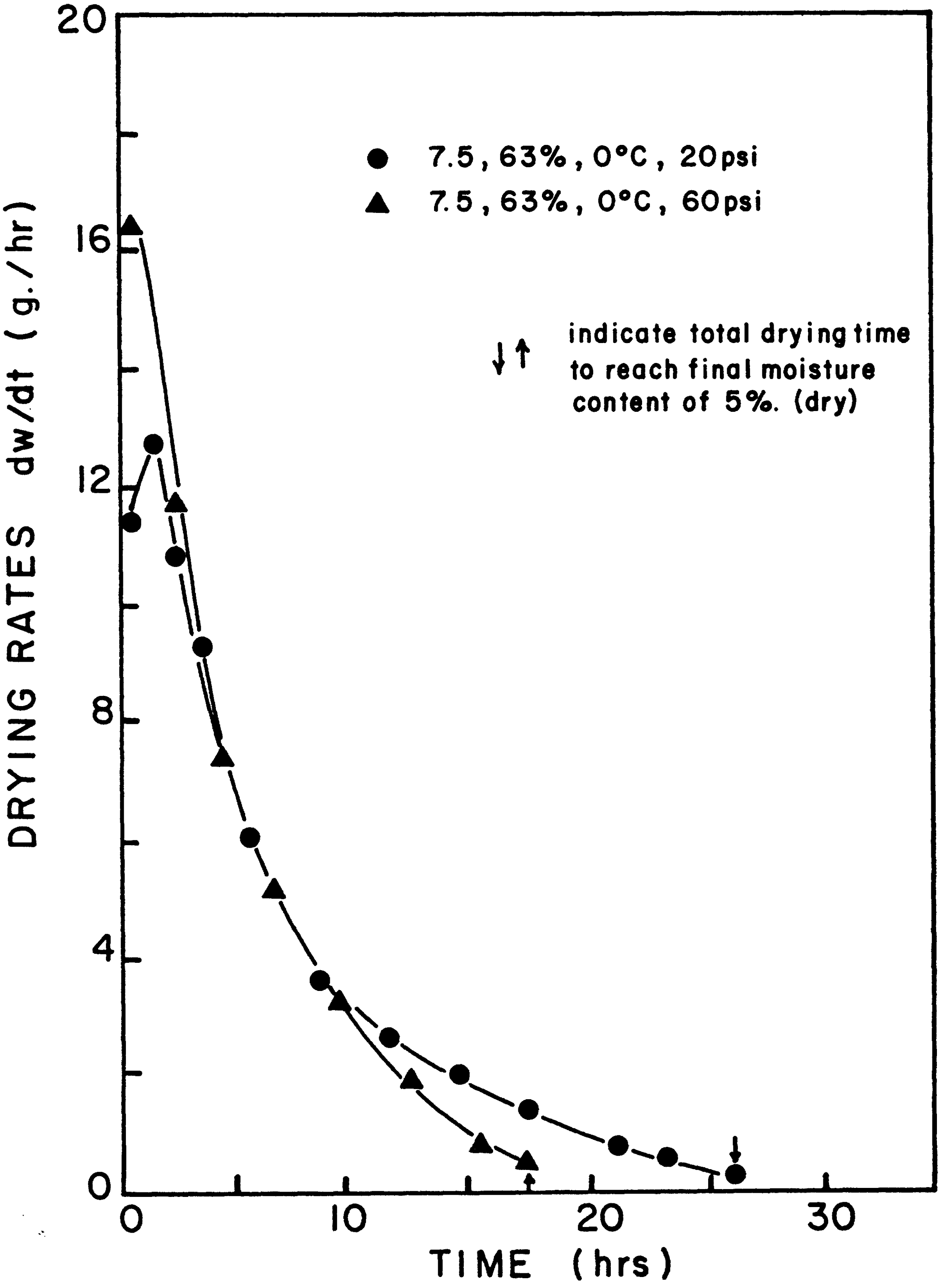
Fig. 4-4. Compression behavior of green beans as a function of time during freeze drying at different levels of compressive pressure.



4.1.2 Dependence of the Drying Rates on Sample Loading (Applied Compressive Pressure)

It was noted that compression during freeze drying results in increased rates of drying which is presumably due to improved heat transfer through the dry layer. It can be expected that there exists a limit to the improvement of the drying rate which can be achieved, since the compression process should result in poorer mass transfer through the dry layer. Thus, at some level of energy input and/or applied compressive pressure, the total process will pass from heat transfer limited to mass transfer limited, which can eventually result in collapse and irreversible structural changes. Figure 4-5 shows a typical example of the effect of increasing the level of applied pressure on drying rates. It can be seen that the sample at the higher level of the applied compressive pressure (60 psi) has higher rates of drying at early stages of the process and reaches the final moisture content in a much shorter time. This effect is even more significant when the drying rate of a sample without any application of compressive pressure (non-compressed) at the same conditions of freeze drying is considered. It was found that during the early stages of the drying, the drying rates of compressed samples were as much as twice as high as those of non-compressed samples (not shown in the figure).

Fig. 4-5. Effect of compressive pressure on drying rates of green beans.



The sharp drop in the drying rates of the samples being compressed during freeze drying is presumably due to the smaller amounts of water left in sample, since most of the water has been sublimated at the early stages of the process. This is indicated by lower drying times to reach a given final moisture content at higher compressive pressures.

4.1.3 Relations of Extents of Drying and Compression Relative to the Applied Compressive Pressure

When the data for compression of green beans during freeze drying is analyzed in terms of volume reduction (percentage change of original height) and extent of drying (percentage of the total water removed), if the removal of a unit of water is associated with a unit fractional increment reduction in volume, a straight line relationship would be expected.

The result of this analysis for various applied pressures is shown in figure 4-6. The data points are given on an hourly basis up to 11 hours of compression/freeze drying. As the drying approaches completion, the data points tend to become quite bunched since the changes in volume and water content are very small. For this reason, data is not presented on an hourly basis after 11 hours. The final degrees of compression are shown at the 100% water removal mark.

In figure 4-6 the effect of compressive pressures of 20 to 60 psi are shown with other process parameters

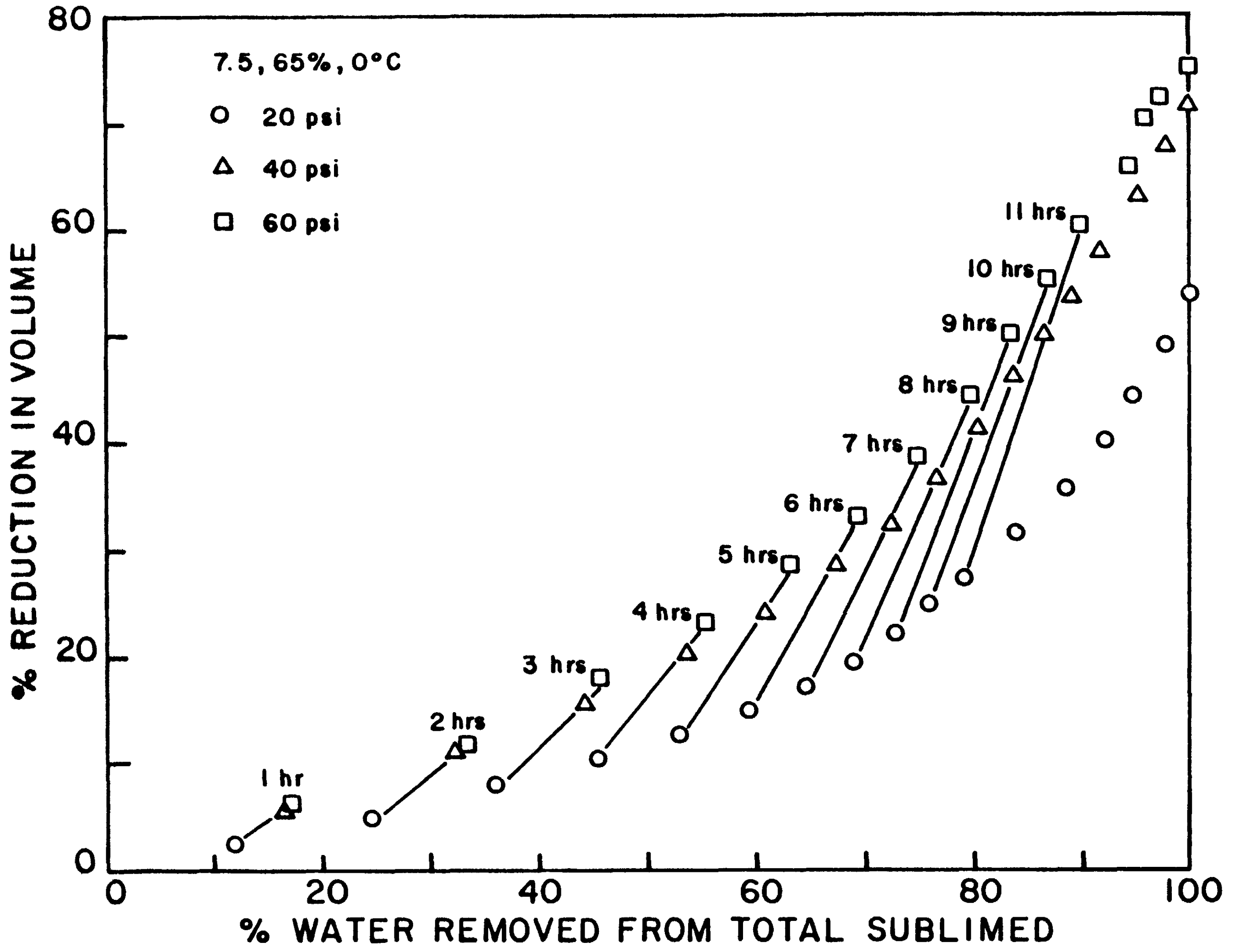


Fig. 4-6. Percent reduction in volume vs. percent water removed during freeze drying of green beans at different levels of compressive pressure.

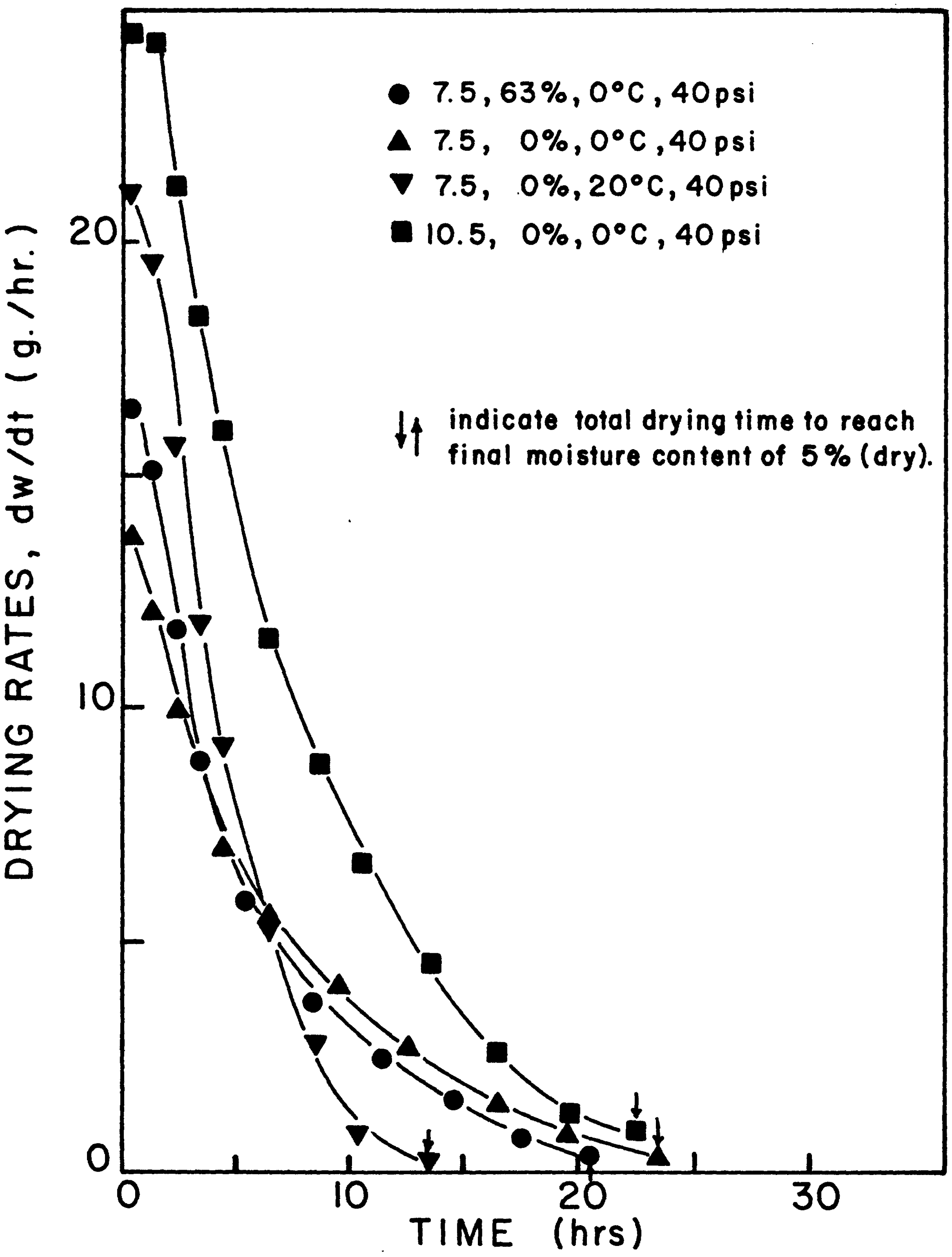
fixed at 7.5 cm, 63%, and 0°C. It can be seen that increased compressive pressures improve both the extent of drying and relative reduction in volume of the sample. However, the increase in volume reduction with increasing compressive pressure seems much greater than that of the water removal, especially at later stages of drying. It is interesting to note that when the data points at each hour are connected for the different compressive pressures applied, a straight line is obtained for each hour as shown in figure 4-6. This indicates that the relative improvement of the extent of drying and compression remains the same as the compressive pressure is increased. This effect, however, seems more significant from 20 psi to 40 psi than 40 psi to 60 psi. The fact that the effect of the increased applied pressure goes mainly into increasing the degree of compression can also be seen from the change in the slope of the straight lines at each hour.

4.1.4 Effect of Heat Input on the Compression and Drying Behavior

Table 4-1 has the summary of the final degrees of compression obtained at different levels of heat input. It appears that for similar experimental conditions but varying heating source temperatures (e.g., set #5; run #15, 10, and 18) where the controlling thermocouples were held at 0°C, 10°C, and 20°C, respectively, there is little effect on the final degree of compression.

Table 4-1 also shows the total drying time for the same experiments being 24-25, 18-19, and 13-14 hours, respectively. The reason for the acceleration of the drying rates due to increased heat input is obvious, as can be seen in figure 4-7 (7.5 cm, 0%, 0°C, 40 psi vs. 7.5 cm, 0%, 20°C, 40 psi). However, close examination of the rate of compression at each temperature setting shows that there is also an increase in the rate of compression at higher temperatures. Figure 4-8 shows the effect of increasing the level of heat input on the extents of drying and compression. It can be seen that all the data points essentially fall on the same path (i.e., the relationship of water removal and volume reduction is the same for all heat inputs). However, at higher temperatures the relative change in both water removal and volume reduction is greater at a given time as indicated by the solid data points at selected times in figure 4-8. For instance, it can be noted that by the end of the 10-hour period, the sample at 20°C has reached final drying and compression, whereas the samples at lower temperatures have not reached the end of the process (figure 4-8). This indicates that the effect of increased heat input mainly accelerates the process, without affecting the final degree of compression at a given applied compressive pressure.

Fig. 4-7. Effect of heat input, cell wall porosity, and size on drying rates of green beans.



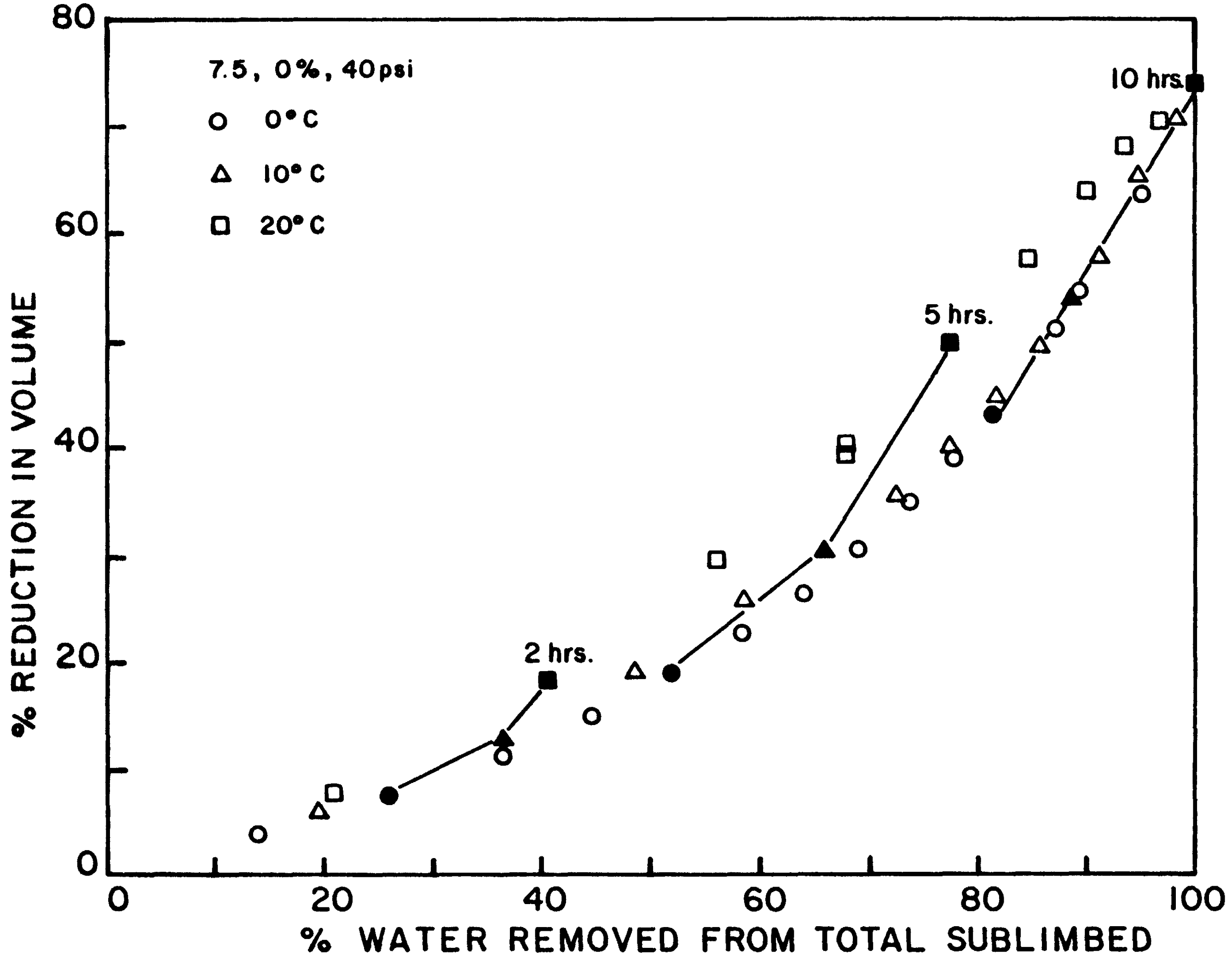


Fig. 4-8. Percent reduction in volume vs. percent water removed during freeze drying of green beans at different levels of heat input.

4.1.5 Effect of Size and Porosity on Compression and Drying Behavior

In a series of experiments, the effect of the porosity of the side walls of the sample cell holder (solid walls 0% vs, 63% open area) was investigated. From table 4-1 it can be seen that there is very little effect of porosity on the final degrees of compression (compare Run #2 and 15, Run #1 and 10, or Run #1 and 19 in Set #5).

As can be expected, higher porosities of the cell wall result in higher drying rates and shorter drying times due to drying from the sides which is shown in figure 4-7 for otherprocess parameters fixed at 7.5 cm, 0°C, and 40 psi.

Similar to the results for porosity, no significant effect was noted in the final degrees of compression for two different sample holder sizes (7.5 x 7.5 cm^2 vs. 10.5 x 10.5 cm^2). This can be seen in table 4-1 by comparing Run #10 with #14, or Run #1 with #9, etc., in set #5.

Figure 4-7 shows the effect of sample holder size on drying rates. As mentioned earlier, the size of the holders were chosen such that the larger sample holder contained twice as much green beans. From figure 4-7 it can be seen that the drying rates for the 10.5 x 10.5 cm^2 sample holder remain about twice as much as for the 7.5 x 7.5 cm^2 sample holder for process condition of 0%, 0°C and 40 psi. It can also be noted that the total drying times to 5% final moisture content for both sizes shown in figure 4-7 are not very different ($\sim$24-25 hours).

Figure 4-9 shows the influence of porosity and sample holder size on extents of drying and compression. Again, variation of these parameters does not appear to change the general path (extent of compression vs. extent of drying) independent of time.

4.2 Compression Behavior of Green Beans. Moisture and Temperature Dependencies as Evaluated with the Instron Universal Testing Machine

In the course of this research, it became apparent that in order to characterize the compression behavior of food materials during freeze drying, a more basic approach was required, since studying compression behavior of foods under freeze drying conditions is very complicated (i.e., interactions of moisture and temperature gradients and other variables) and is also a very slow process (each data point requires a complete freeze drying experiment under controlled conditions).

It is apparent that deformation behavior of food materials depends on their mechanical properties, the values of which seem to be a strong function of moisture content and temperature.

In order to determine the basic compressive mechanical properties relevant to compression behavior during freeze drying and the stress-strain behavior of this product as a function of moisture and temperature, a series of compression studies were conducted with the Instron Universal

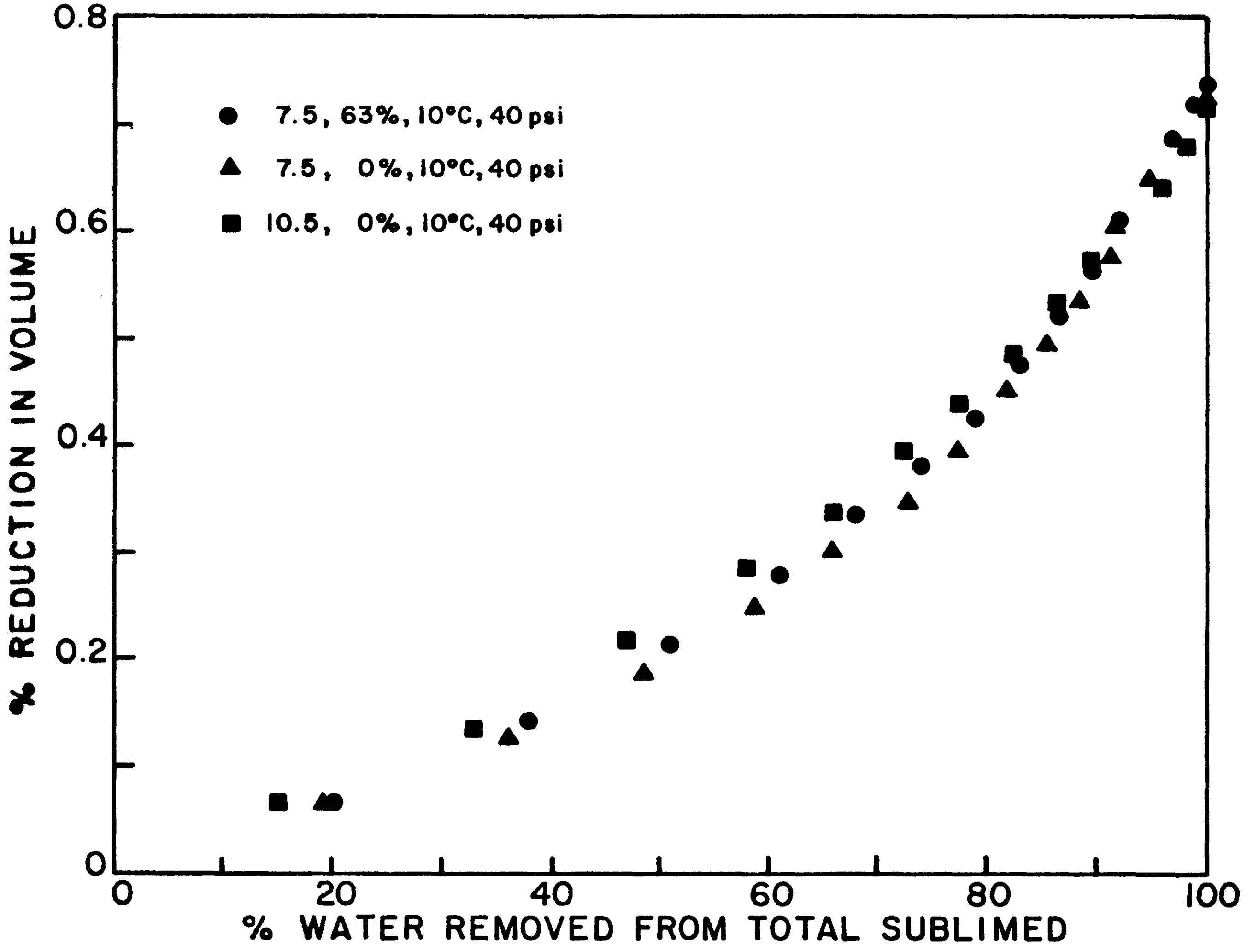


Fig. 4-9. Percent reduction in volume vs. percent water removed during freeze drying of green beans at different sample holder sizes and wall porosities.

Testing Machine. When mechanical stress is applied to food materials (and other polymeric compounds), there exists a critical range of temperature and moisture content for which the solid structure can undergo deformation without irreversible structural changes. In this research a detailed study of the influences of these variables (moisture and temperature) on mechanical properties of green beans (freeze-dried initially) as related to compression of this product was conducted. The results were analyzed in terms of these two variables (moisture and temperature) independently and interdependently and further related to the mechanical properties of this product.

4.2.1 Effect of Moisture and Temperature on Compressibility of Freeze-Dried Green Beans

Freeze-dried green beans rehumidifed to different moisture contents [up to about 45% moisture content (dry basis)] and adjusted to different temperatures (+24°C to -25°C) were compressed uniaxially in the Instron Universal Testing Machine and the force-deformation relationship was determined. From the force-deformation curves, the stresses requied to achieve particular strains were determined for samples of varying moisture contents and temperatures.

Figures 4-10 through 4-13 show the relationships of applied stress and moisture content (Instron-derived, stress-moisture curves) to achieve particular strains at given sample temperatures with a cross-head speed of

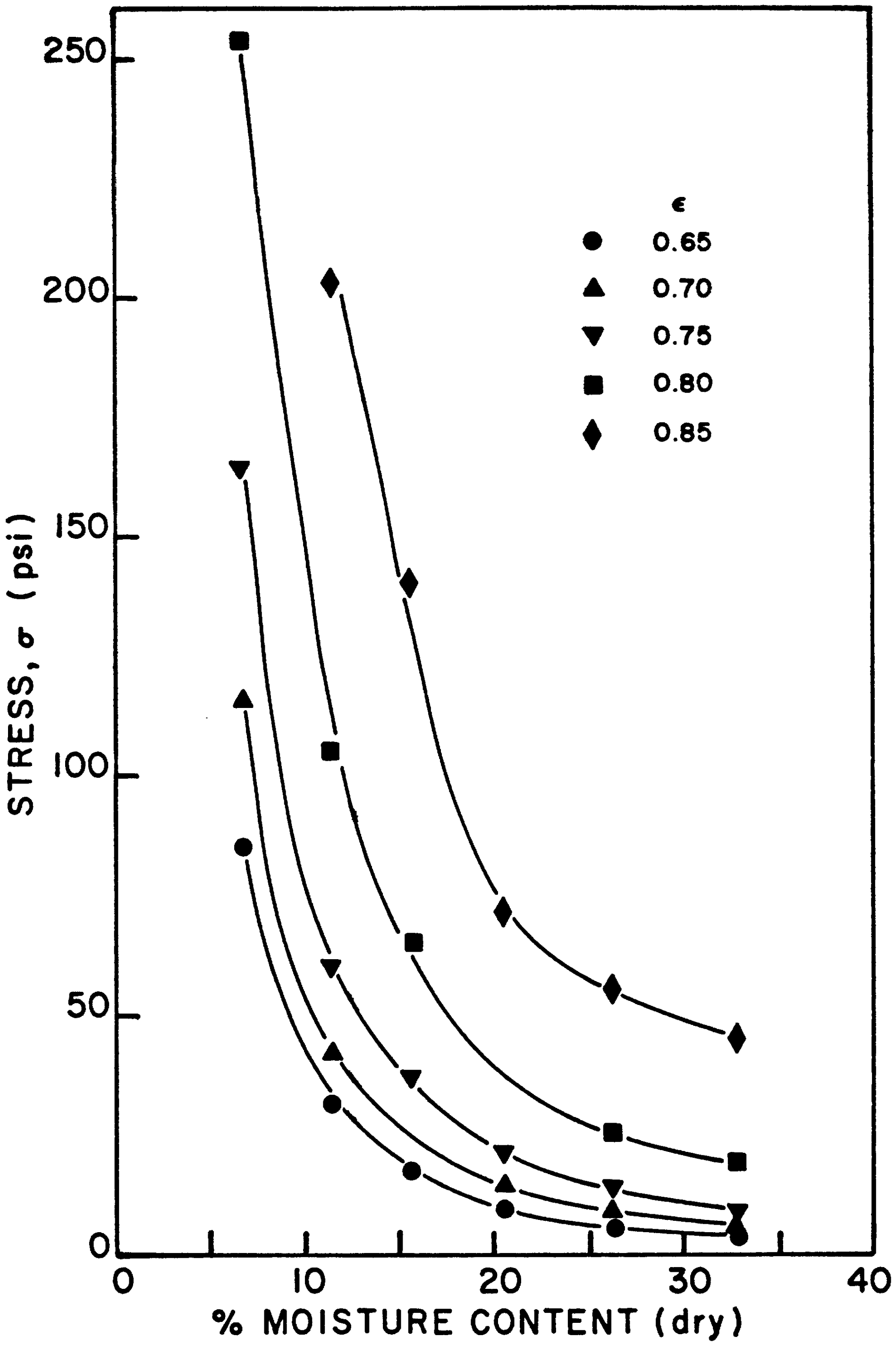


Fig. 4-10. Effect of moisture content on stress to achieve given strains at +24°C (Instron tests).

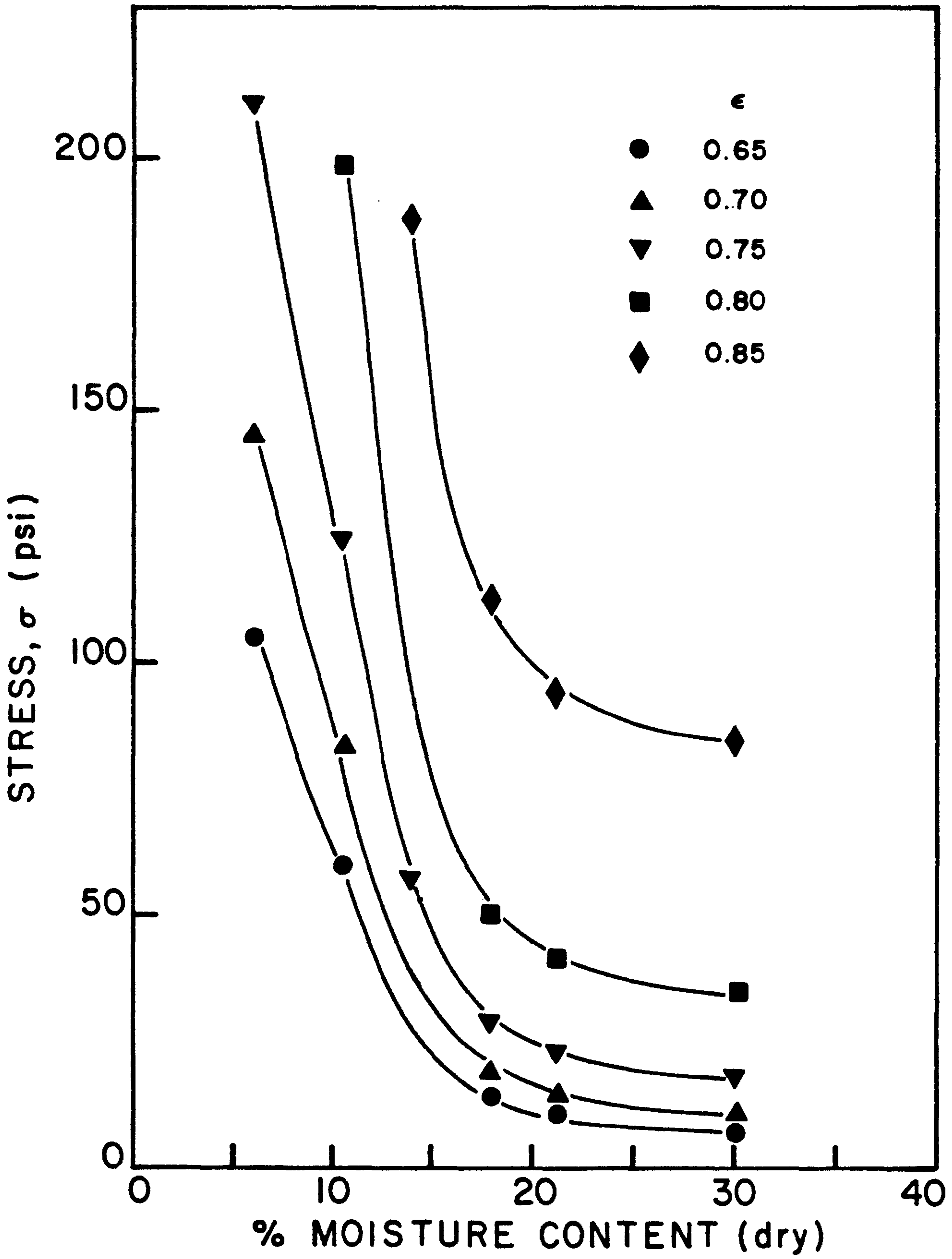


Fig. 4-11. Effect of moisture content on stress to achieve given strains at +15°C (Instron tests).

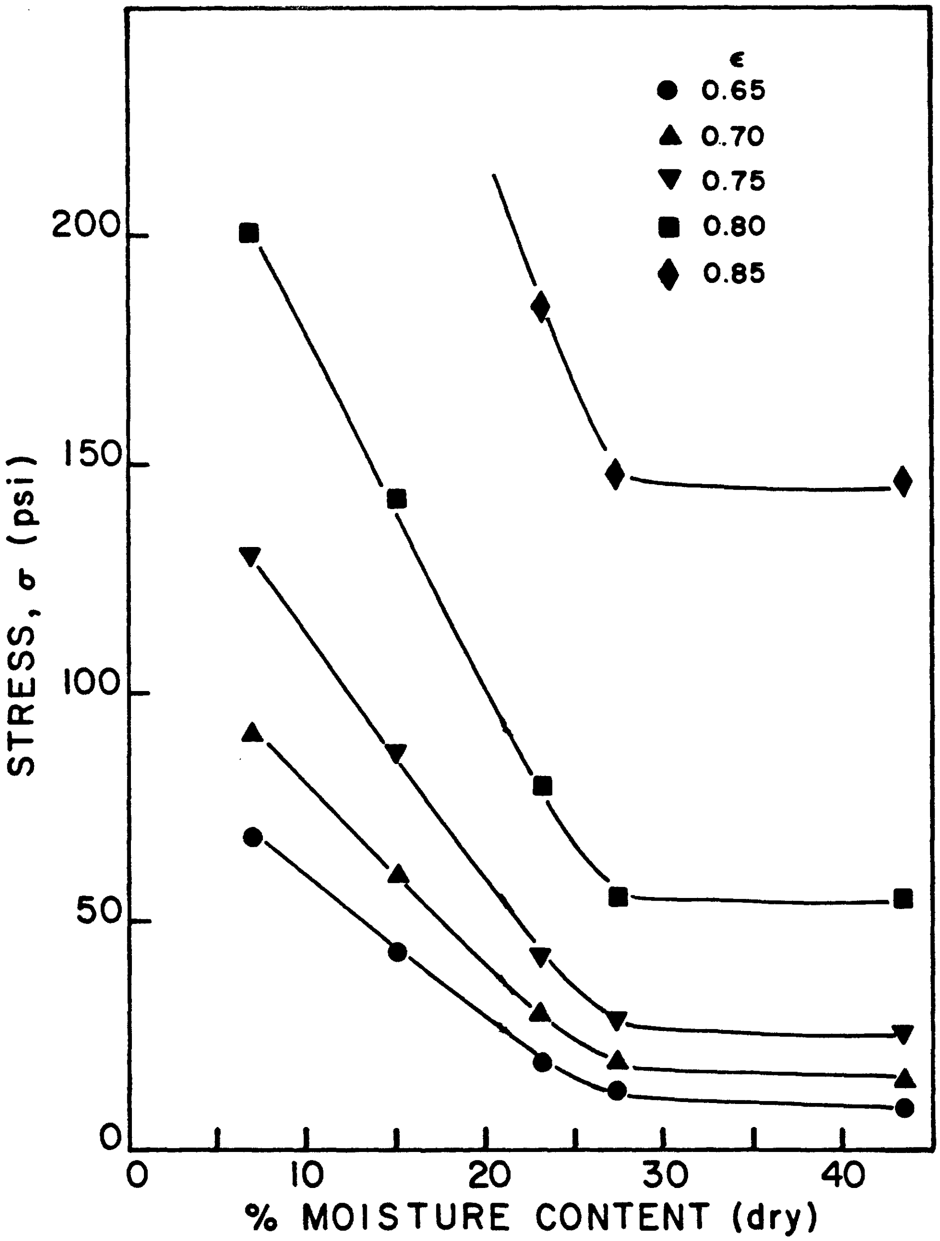


Fig. 4-12. Effect of moisture content on stress to achieve given strains at -10°C (Instron tests).

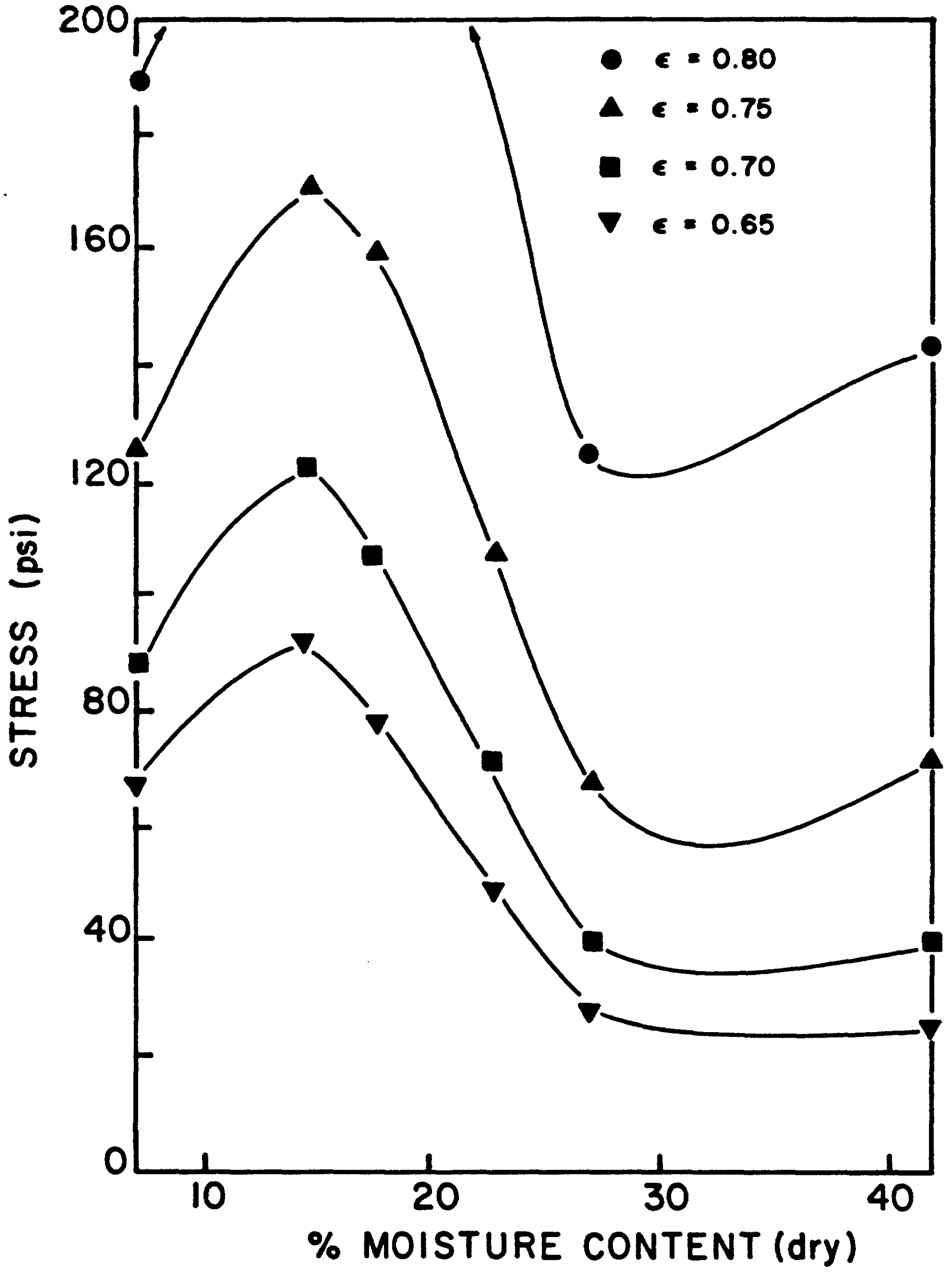


Fig. 4-13. Effect of moisture content on stress to achieve given strains at -25°C (Instron tests).

20 mm/min. Figure 4-14 shows the stress-moisture content relationship for producing a strain of 0.75 for samples of varying moisture contents and temperatures.

It can be seen that the general compressive behavior for green beans can be described as a decrease in forces or stresses required to produce a fixed volume reduction or strain as the moisture content increases, until the moisture content reaches the range of 20%-35%, at which point moisture content shows little further influence.

The effect of temperature can be better visualized in figure 4-14. If the curve at -25°C is not considered, the influence of temperature on the stresses to achieve a given strain ($\varepsilon = 0.75$ in this case) at a given moisture content is similar (i.e., lower temperature requires more stress), though the differences are less significant than those for varying the moisture content. It should be noted that at -25°C most of water is in the solid state which makes the material quite brittle. This phase transition (water to ice) can affect the validity of the stress-strain relationship at this temperature relative to other temperatures tested.

The observed effects of increasing moisture content and temperature on compressibility of the product are expected as described earlier in section 2.5 and can be attributed to the decrease in relative rigidity of the food structure.

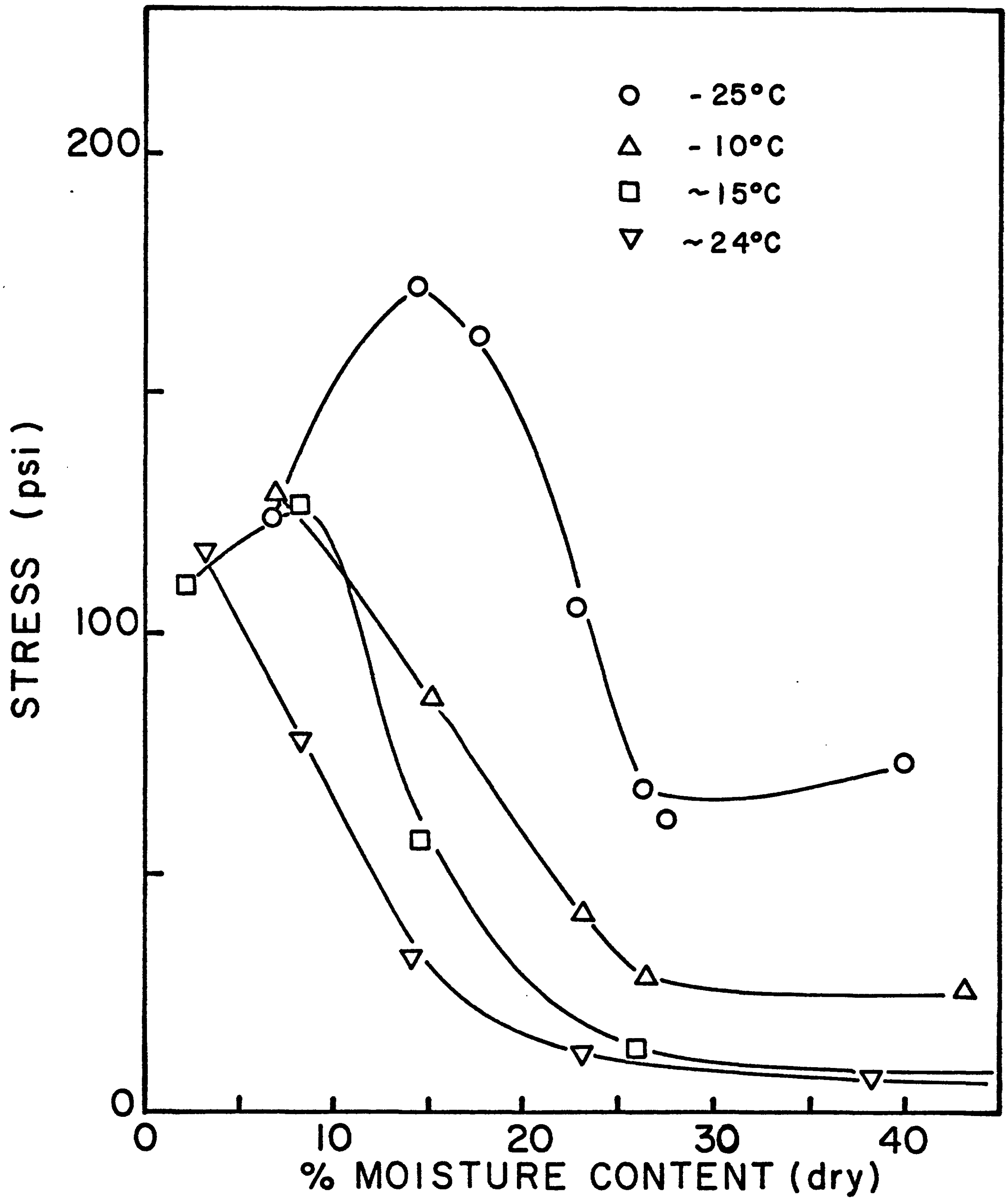


Fig. 4-14. Effect of moisture content on stress to reach a strain of 0.75 at different temperatures (Instron tests)

4.2.2 Effect of Cross-Head Speed or Strain Rate on Compressibility

In order to test the effect of the strain rate on compresibility, freeze-dried, rehumidifed green beans at various levels of moisture content were compressed uniaxially at room temperatures using the Instron Machine at a cross-head speed of 50 mm/min. Figure 4-15 shows the stresses required to achieve given strains for both cross-head speeds of 20 mm/min and 50 mm/min at room temperature (+24°C). It can be noted that the results are very similar and there is no apparent effect of the strain rate at least for the two rates tested. This would indicate that at these two different levels of cross-head speed the samples probably did not have any time for stress relaxation during the compression process that could result in deviation in the force-deformation response. As for the compression during freeze drying experiments, although the strain rates were very slow (∿1 cm/5- 10 hrs), however, due to constant application of compressive pressure for the duration of the experiment, it can be expected that the compression of any region in the dry layer was instantaneous with no time for stress relaxation.

4.2.3 Effect of Moisture and Temperature on Mechanical Properties

Figures 4-16 through 4-18 illustrate the effect of moisture and temperature on mechanical properties of green

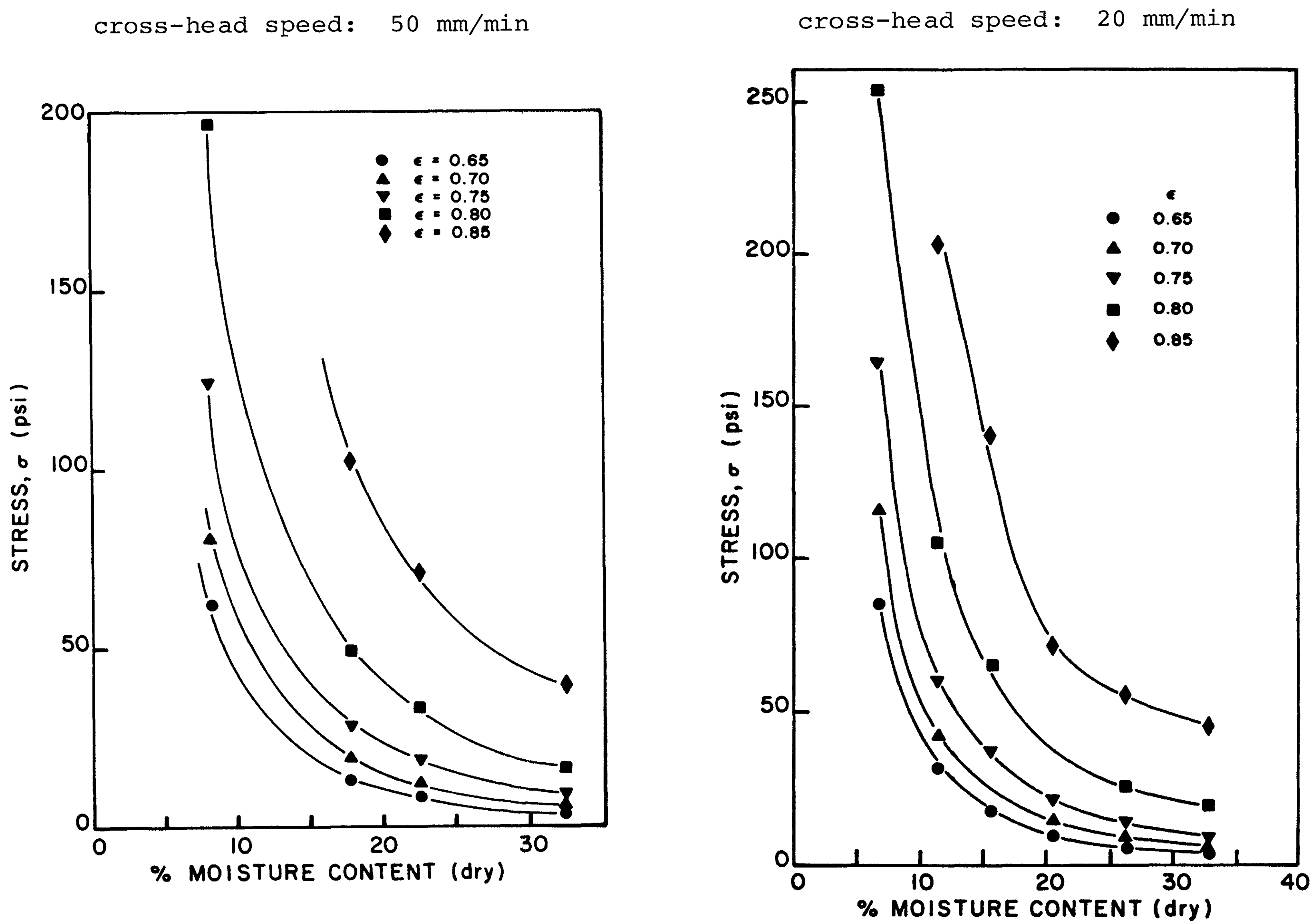


Fig. 4-15. Effect of moisture content on stress to achieve given strains at different cross-head speeds (+24°C, Instron tests).

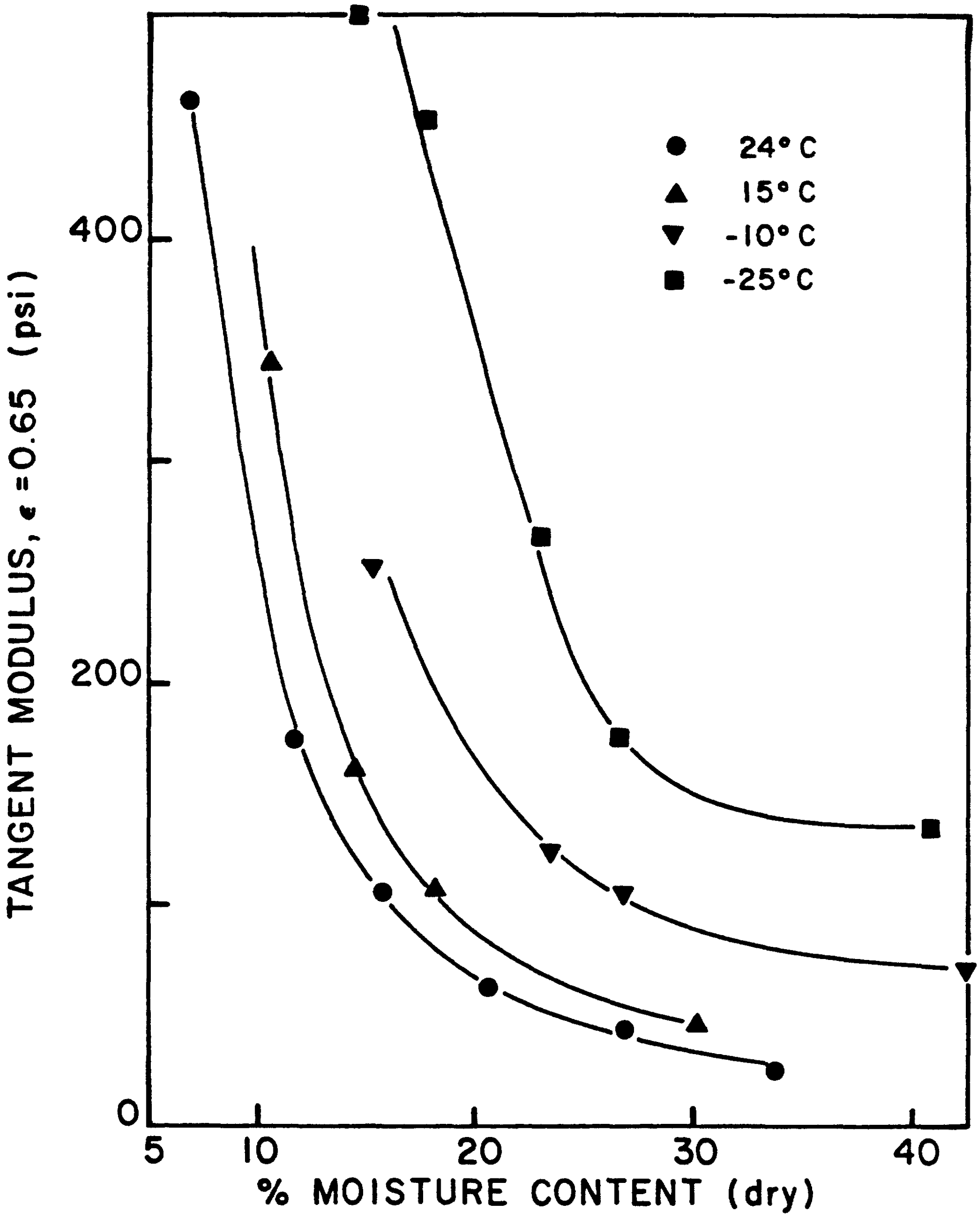


Fig. 4-16. Effect of moisture content (and temperature) on tangent modulus calculated at strain (ε) = 0.65 (Instron tests).

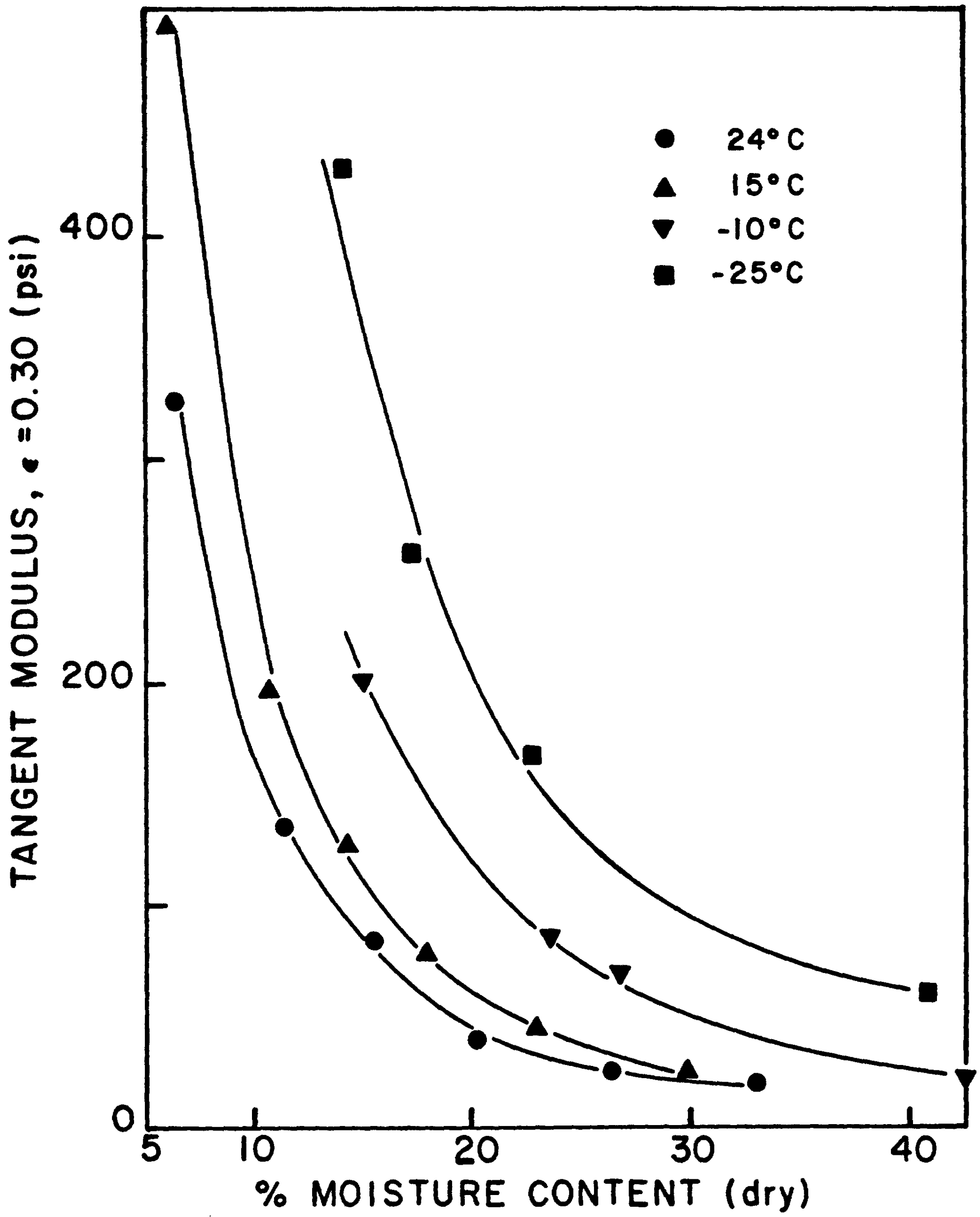


Fig. 4-17. Effect of moisture content (and temperature) on tangent modulus calculated at strain (ε) = 0.30 (Instron tests).

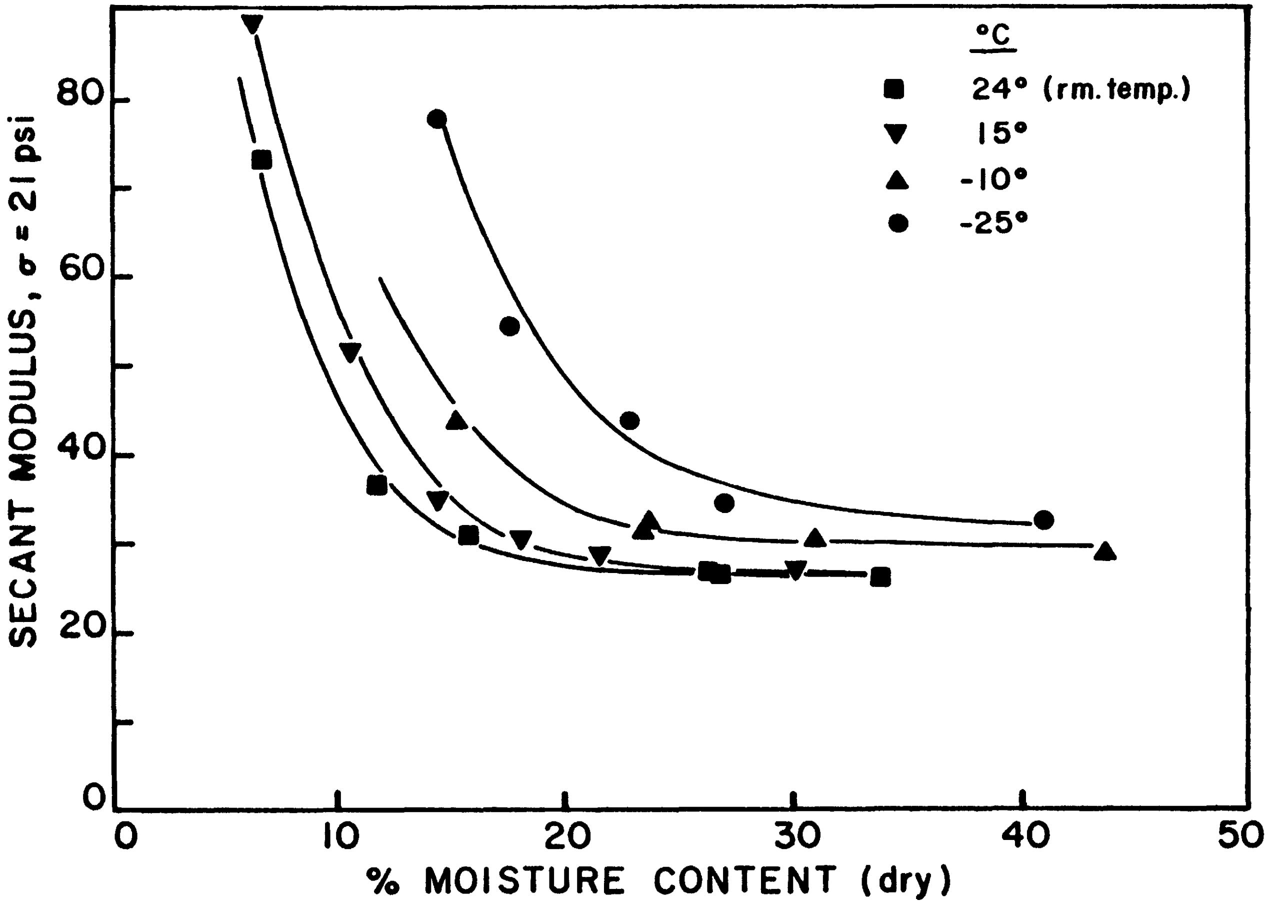


Fig. 4-18. Effect of moisture content (and temperature) on secant modulus calculated at stress (σ) = 21 psi (Instron tests).

beans. The mechanical properties which were measured are tangent modulus (σ/ε) calculated at two different strains (0.65 and 0.30) (figures 4-16 and 4-17 respectively), and secant modulus (σ/ε) calculated at a stress of 21 psi (figure 4-18). As expected, it can be noted that the effect of increaseing both moisture and temperature causes a reduction in the values of these mechanical properties both of which represent relative rigidity of the material.

4.2.4 Moisture-Temperature Dependencies: Common Mechanical Properties and Similar Compression Behavior (Stress-Strain Relationship)

The relative effects of moisture and temperature on compressibility of freeze-dried, rehumidifed green beans suggest the existence of specific combinations of these two variables which will result in common values for mechanical properties of the food and in a similar compression response under compressive stress.

In fact, similar stress-strain relationships are obtained for freeze-dried green beans having specific combinations of moisture and temperature which correspond to common values of the mechanical properties investigated. Figures 4-19 through 4-21 present the moisture-temperature relationships for specified values of tangent modulus and secant modulus which give similar stress-strain relationship for freeze-dried green beans. It should be noted that each individual curve from the family of curves

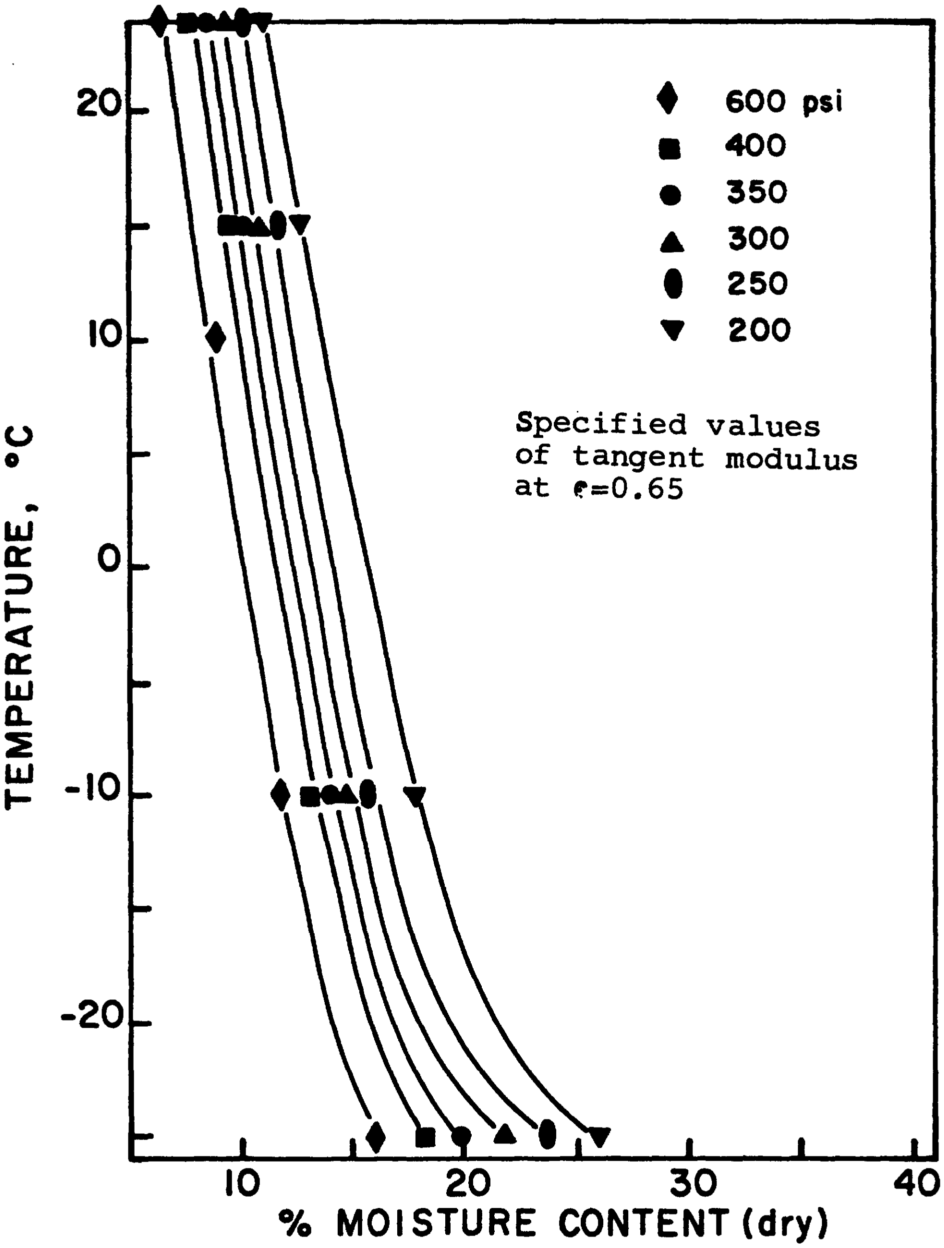


Fig. 4-19. Moisture-temperature relationship with equivalent compression behavior at specified values of tangent modulus (at ε = 0.65) obtained in Instron tests.

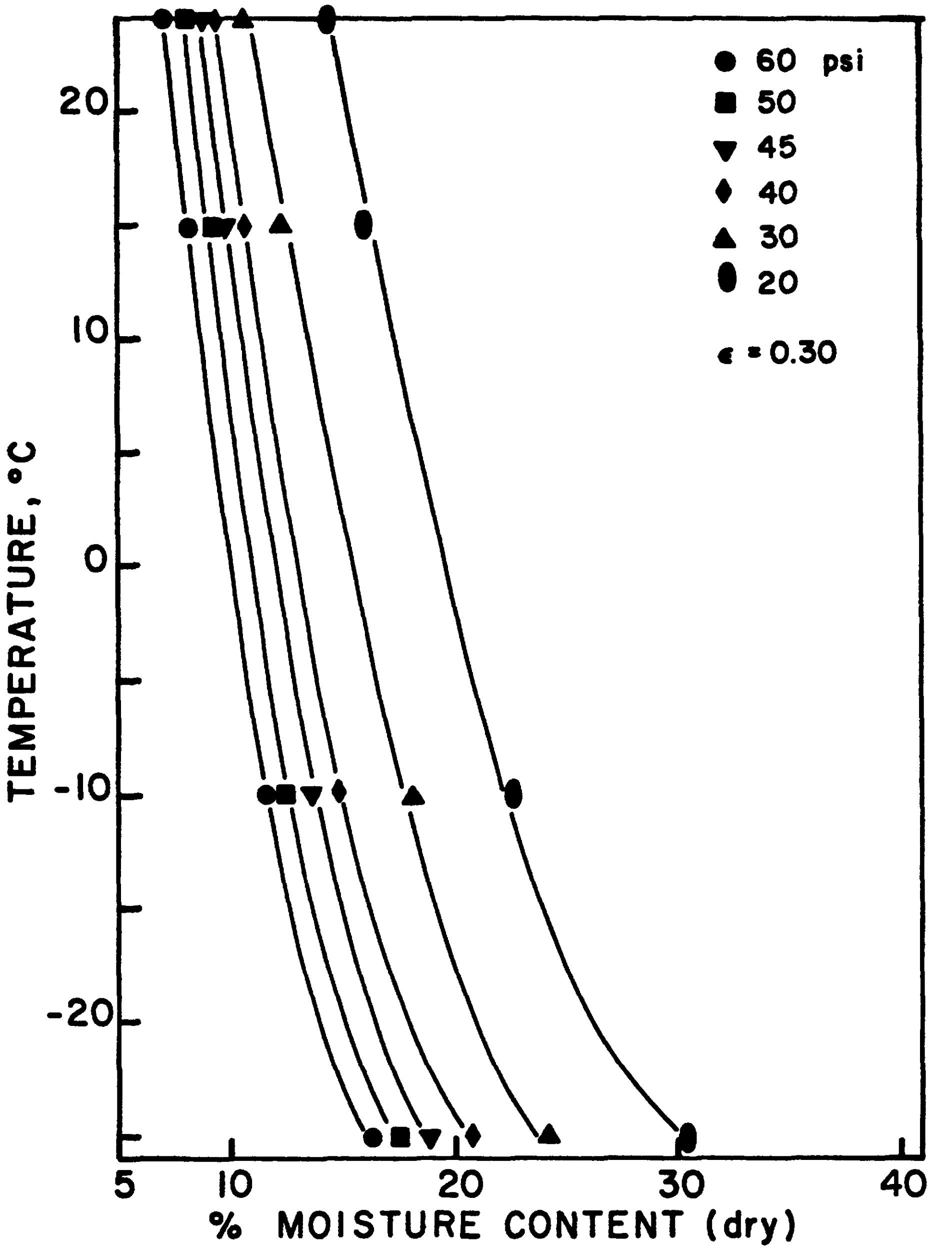


Fig. 4-20. Moisture-temperature relationship with equivalent compression behavior at specified values of tangent modulus (at $\varepsilon = 0.30$) obtained in Instron tests.

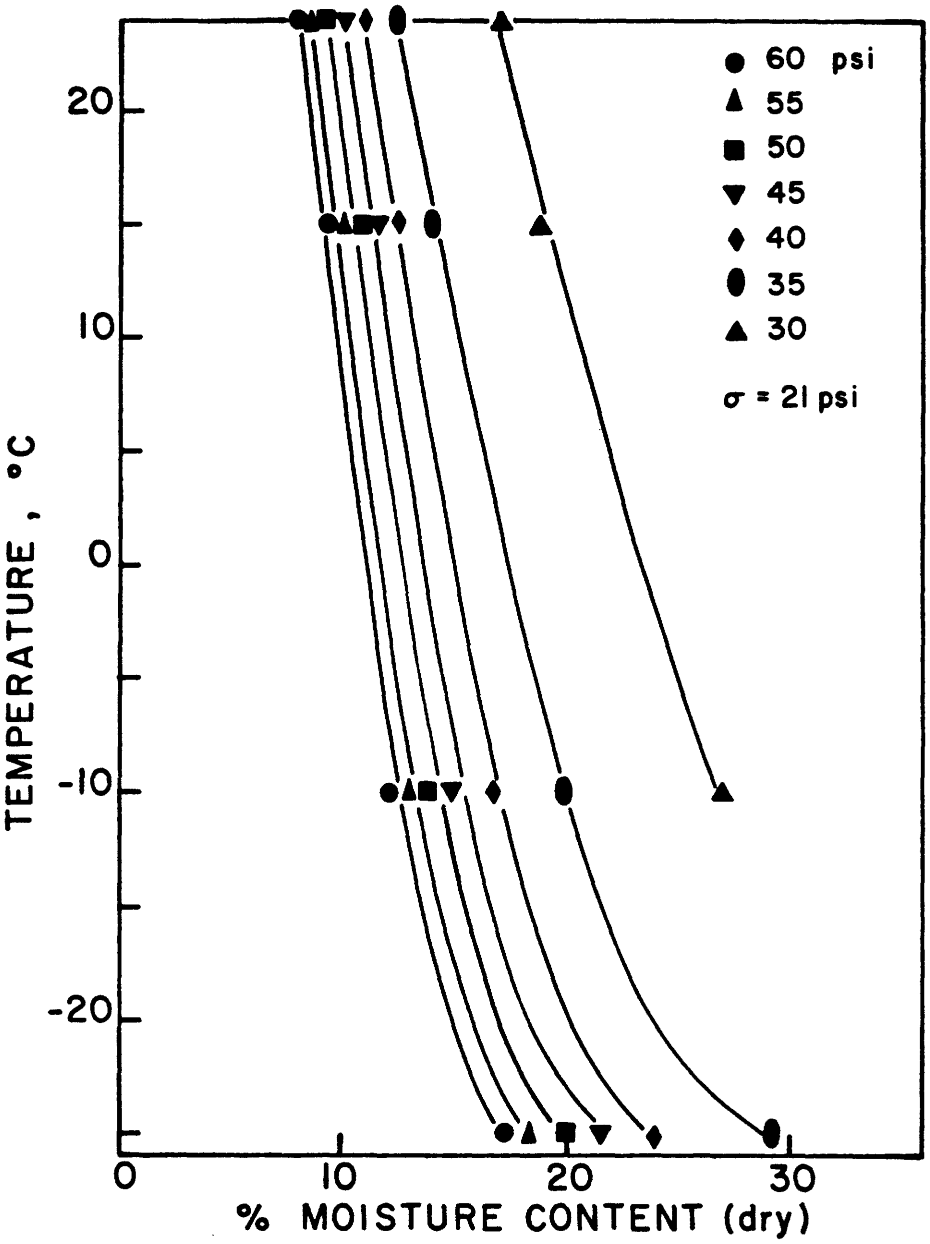


Fig. 4-21. Moisture-temperature relationship with equivalent compression behavior at specified values of secant modulus (at σ = 21 psi) obtained in Instron tests.

presented corresponds to a specified value of a given mechanical property, and further all the moisture-temperature combinations which fall on each individual curve (i.e., have a specified mechanical property) have similar compression behavior (stress-strain relationship).

Based on the results presented in Figures 4-10 through 4-13 and figures 4-19 through 4-21, it is possible to calculate compressive behavior of freeze-dried green beans which have been rehumidifed and adjusted to any combination of temperature and moisture content. Given either a value of moisture and temperature or a value of a pertinent mechanical property, it is possible to predict and construct a stress-strain relationship for freeze-dried green beans. This is described further in section 4.3.

4.3 Correlation of Compression Behavior during Freeze Drying to Compression Behavior with the Instron Universal Testing Machine

4.3.1 Moisture-Temperature Relationships and Mechanical Properties of the Dry Layer during Freeze Drying

According to the results presented in section 4.2 and literature information, it can be stated that for a given food material, the mechanical properties at any location in the dry layer of the freeze-drying sample will depend on the local temperature and moisture content. Considering moisture and temperature gradients that occur in the dry layer during freeze drying, it can be expected that there exist various combinations of moisture and temperature which will give mechanical properties suitable for compression during freeze drying.

If it is assumed that the same factors which govern compressive behavior of the food in the Instron Universal Testing Machine also apply to the compressive behavior during freeze drying, it is then possible to relate the two processes. To achieve this, it is first necessary to consider the temperatures and moistures occurring in freeze drying.

As reported earlier (section 2.8), Gentzler and Schmidt (1973) showed that if the temperature of the frozen region during freeze drying process remains constant, then at any instant in time, the temperature and moisture content of any location in the dry layer zone are fixed. Furthermore, for each given temperature in the dry layer, there will be a unique value of moisture content at that location whose value can be determined from figure 2-11 for skim milk and granulated coffee. The estimated moisture and temperature relationship in the dry layer for green beans during freeze drying at a constant ice region temperature of -25°C is shown in figure 4-22.

It was shown earlier that for any given value of moisture and temperature, there will be a fixed value of each mechanical property, whose value for tangent modulus and secant modulus can be estimated from figure 4-19 through 4-21. Thus, given the moisture-temperature relationship

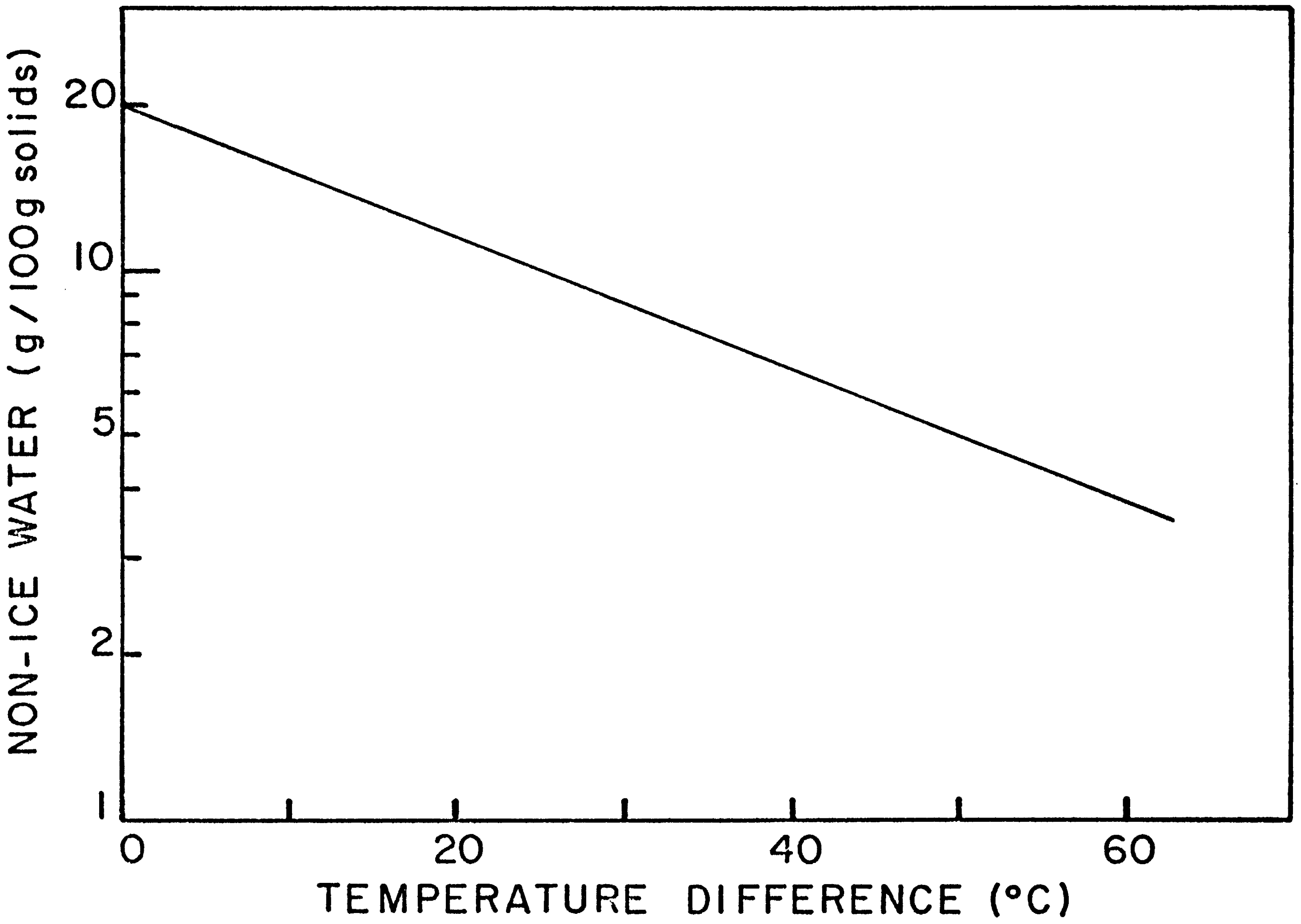
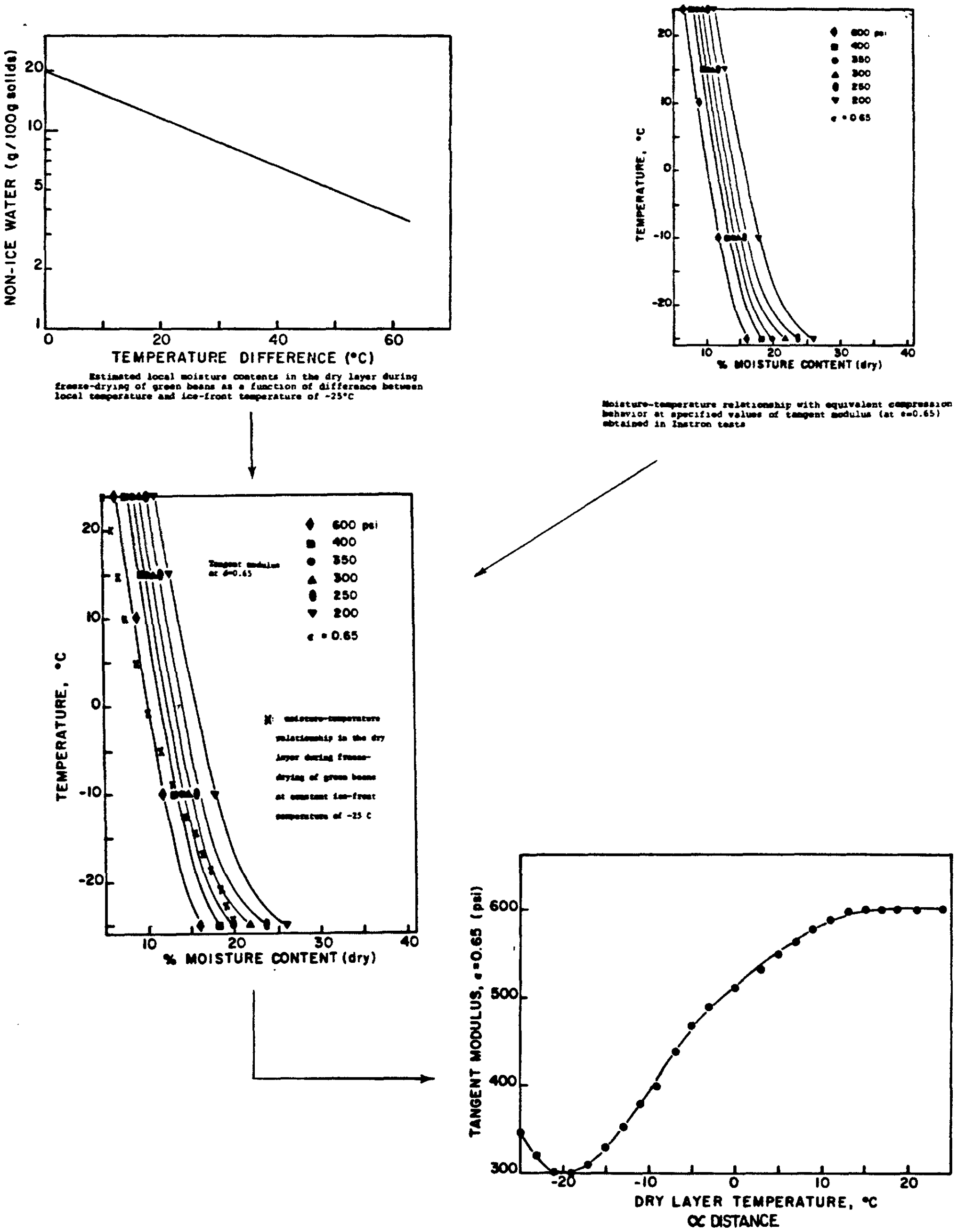


Fig. 4-22. Estimated local moisture contents in the dry layer during freeze drying of green beans as a function of difference between local temperature and ice-front temperature of -25°C.

that exists in the dry layer during freeze drying (figure 4-22), it is possible to calculate the range of the values of the given mechanical property in the dry layer during freeze drying. As an example, figure 4-23 shows how this can be done for tangent modulus at $\varepsilon = 0.65$ by combining figures 4-19 and figure 4-22 and assuming an ice-front temperature. In figure 4-23 fixed values of temperatures with corresponding values of moisture contents in the dry layer during freeze drying (from figure 4-22) are transferred to figure 4-19 which represents moisture-temperature relationship at specified values of tangent modulus (ε=0.65) obtained in Instron studies. Combination of these two provides the values of the given mechanical property at each location of the dry layer corresponding to those local moistures and temperatures. By knowledge of the values of the mechanical property at each location in the dry layer, it is possible to construct the range of the mechanical property as a function of dry layer temperature or distance from the ice interface which is shown in the bottom of figure 4-23 for tangent modulus at $\varepsilon = 0.65$. Figure 4-24 and 4-25 show the estimated range of tangent modulus at $\varepsilon = 0.30$ and secant modulus at $\sigma = 21$ psi as a function of dry layer distance. It should be noted that in these figures location of the <u>minima</u>, which is close to but not adjacent to the ice-front, indicates the most compressible region that exists in the dry layer at any instant in time. This implies that if a constant



Estimated local moisture contents in the dry layer during freeze-drying of green beans as a function of difference between local temperature and ice-front temperature of -25°C

Moisture-temperature relationship with equivalent compression behavior at specified values of tangent modulus (at ε=0.65) obtained in Instron tests

Range of tangent modulus (ε=0.65) that exist in the dry layer during freeze drying of green beans

Fig. 4-23. Estimated values of tangent modulus (at $\varepsilon = 0.65$) in the dry layer during freeze drying of green beans as a function of distance in the dry layer.

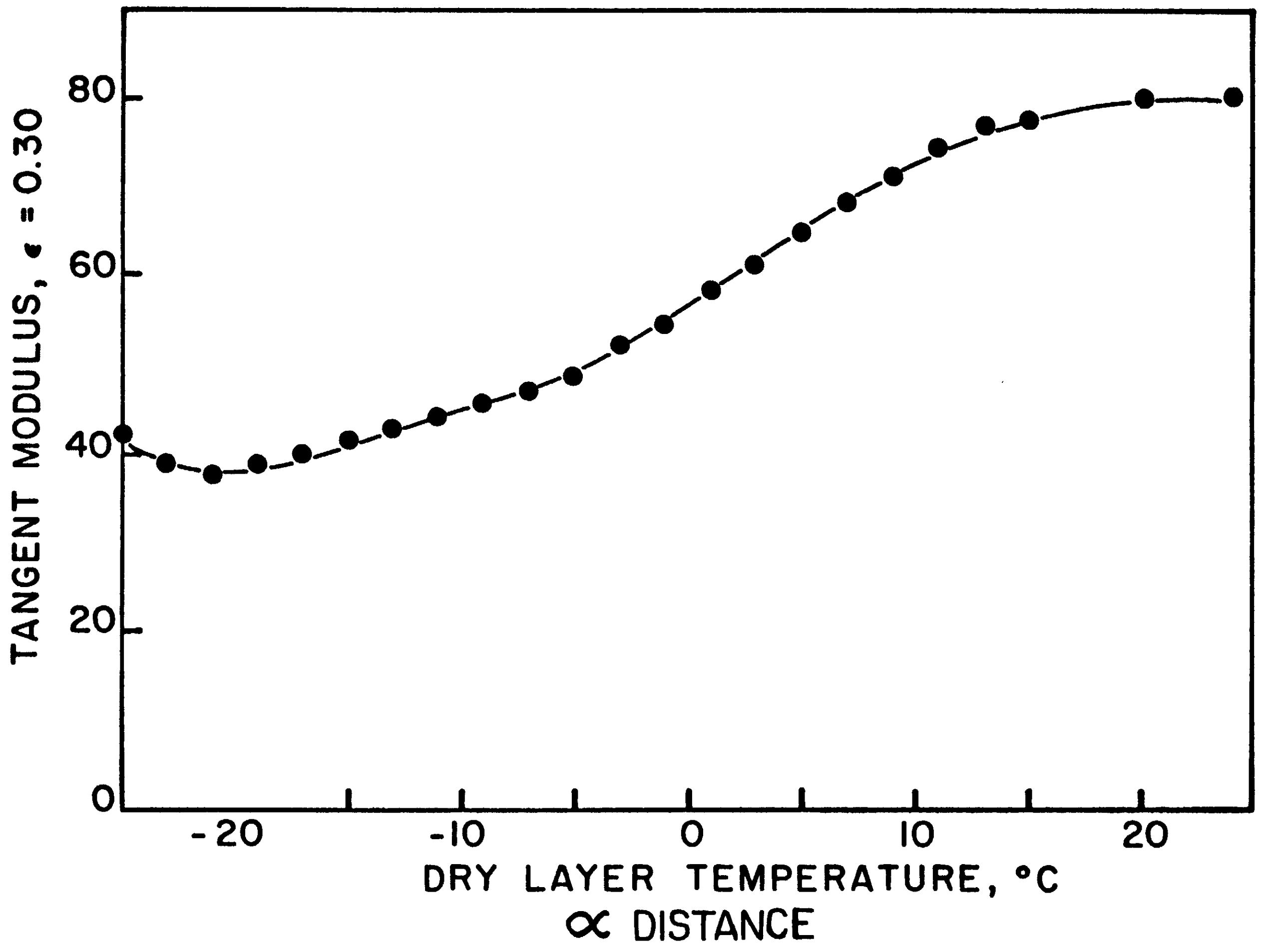


Fig. 4-24. Estimated range of tangent modulus (at ε = 0.30) that exist in the dry layer during freeze drying of green beans.

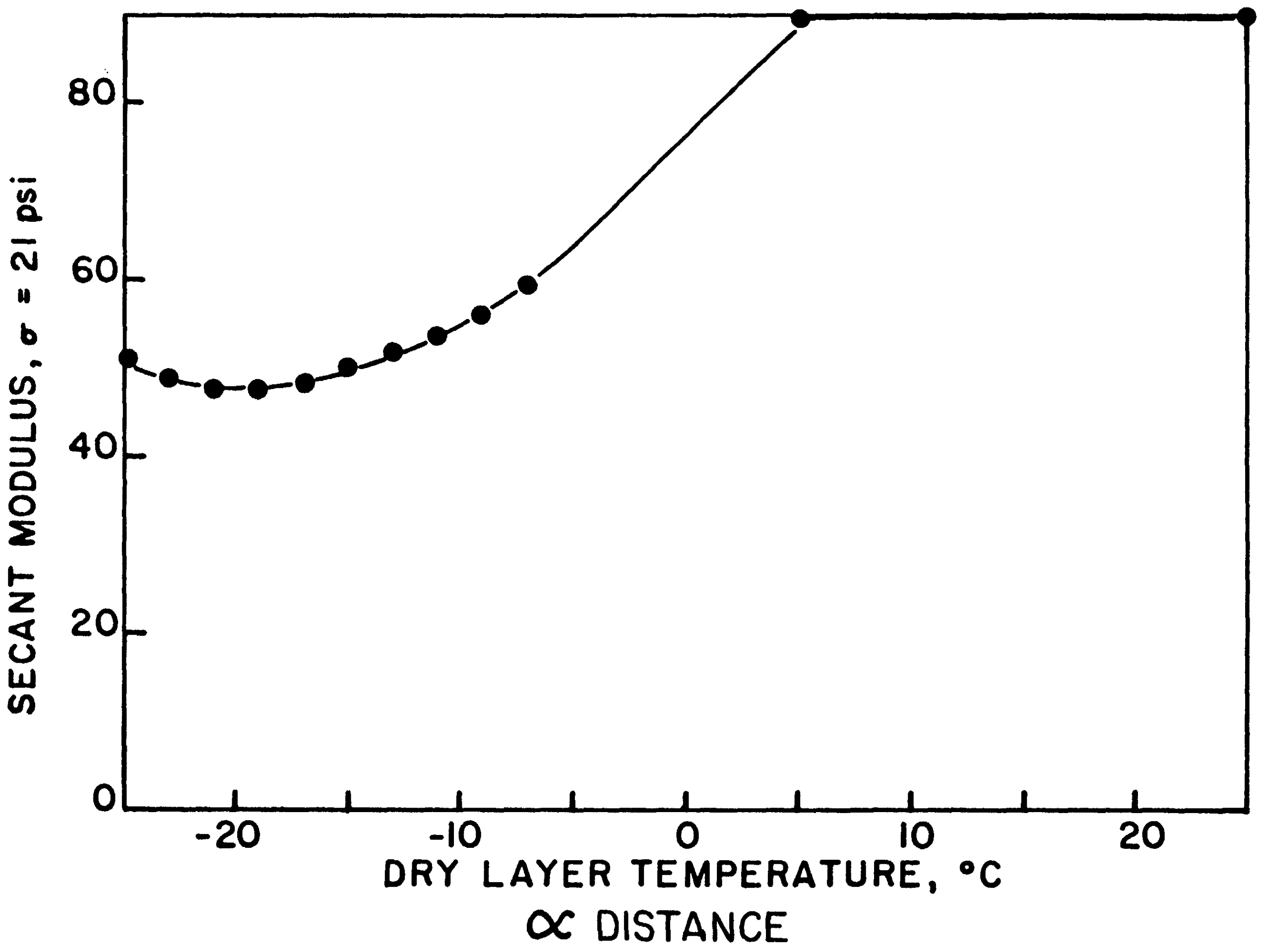


Fig. 4-25. Estimated range of secant modulus (at σ = 21 psi) that exist in the dry layer during freeze drying of green beans.

compressive pressure is applied to the sample at any instant in time during freeze drying, each location in the dry layer will undergo a deformation (compression), the degree of which depends on the value of the mechanical property of that location relative to the applied compressive pressure. Furthermore, the highest degree of compression will occur in the location with minimum value of modulus.

Figure 4-26 shows the behavior of the modulus as a function of time and distance. Assuming a constant ice-front temperature means that the value of the minima does not change with time; however, its location moves inward as the ice front recedes, except for the initial and final stages of the drying process. Initially the drying surfaces are very cold resulting in high values of the modulus (high rigidity). At the end of the drying process when ice disappears and the moisture gradients drop, the minima also disappear resulting in a rigid structure again. It is interesting to note that the rotation of the gradient that occurs about the constant surface temperature produces decreasing values for the temperature gradient, and it is thus expected that the distance of the minima from the ice-front does not stay constant as drying proceeds. This is shown in figure 4-26.

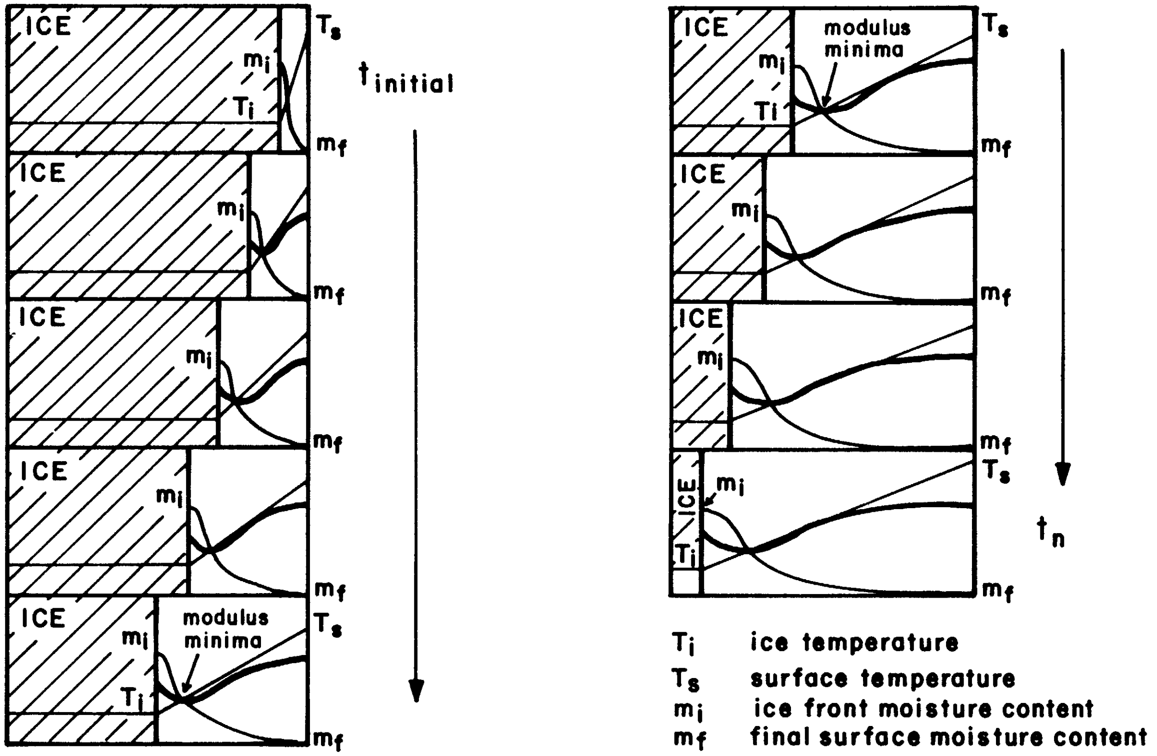


Fig. 4-26. Behavior of modulus with respect to time and location during freeze drying at constnat ice-front temperature.

4.3.2 Mechanism of Compression during Freeze Drying at Constant Applied Compressive Pressure

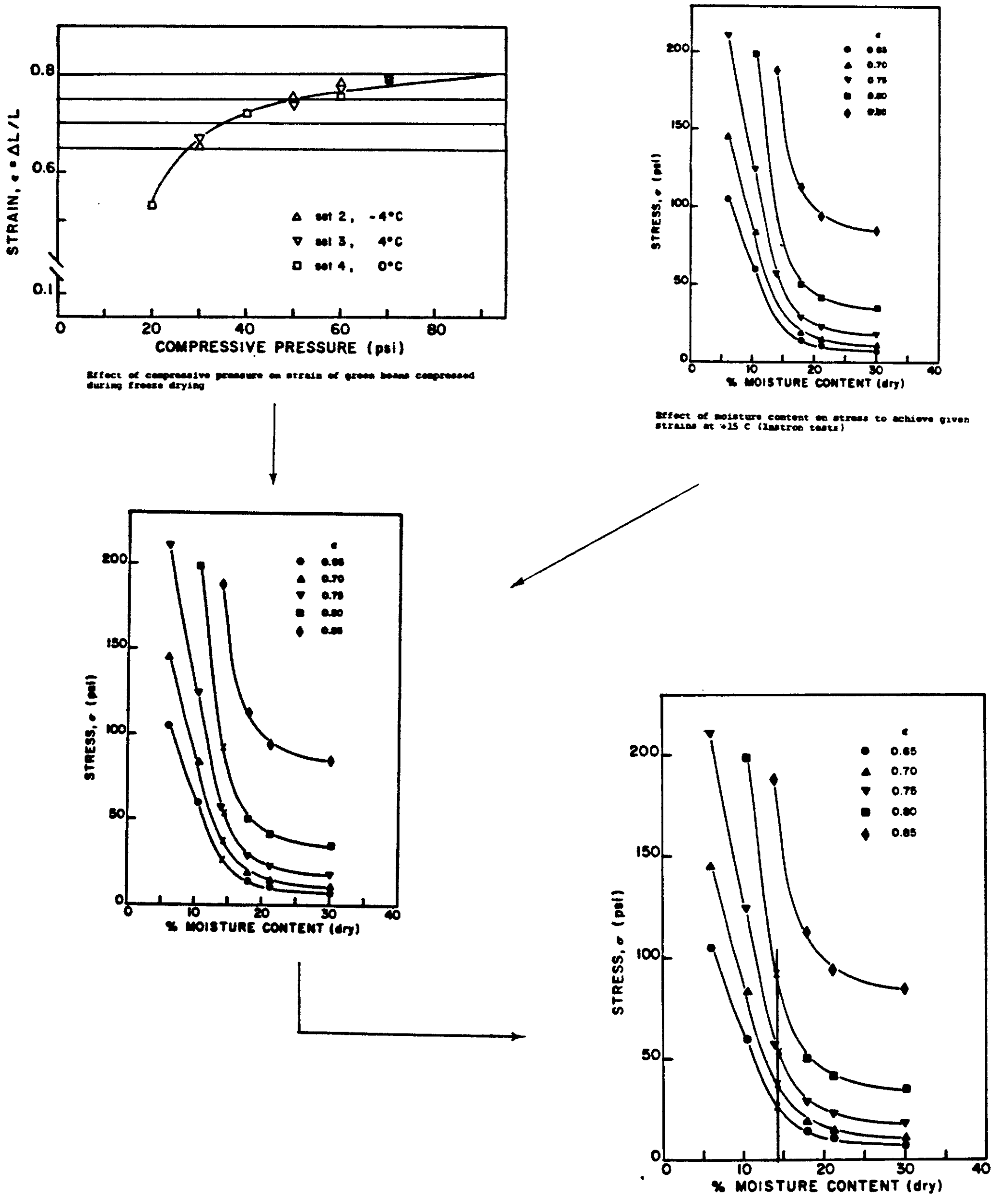
It was shown earlier that from knowledge of the value of the given mechanical property and the applied compressive pressure, the degree of compression that will occur can be determined. Thus considering the behavior of the modulus in the dry layer at any instant in time at a constant applied compressive pressure, we note that the dry layer will undergo a certain deformation, the degree of which can be determined at each location in the dry layer from the value of the modulus at that location and time. As mentioned before, the maximum compression will always occur at the location corresponding to minimum rigidity in the dry layer, and thus, the minimum value of the modulus.

It can be expected that, since the minima moves inward through the dry layer with time (figure 4-26) with a constant value, all the locations in the dry layer will eventually undergo a fixed maximum compression which depends on the minimum value of the modulus, and the given applied compressive pressure. This will result in a fixed compression throughout the sample by the end of the compression/freeze drying process.

4.3.3 Development of Means to Predict Compression Behavior during Freeze Drying; Compressive Pressure-Strain Relationship

Considering that if moisture and temperature of an already freeze-dried sample are independently adjusted so that the sample has a value of the modulus which corresponds to the minimum of the modulus that exists in the dry layer during freeze drying, it can be expected that both samples will have equivalent compression behavior for the same applied stress. In other words, the previously freeze-dried sample with adjusted uniform temperature and moisture content will have the same mechanical properties as that location in the dry layer of the freeze drying sample having minimum value of the modulus.

In fact when compressive pressure-strain data for compression during freeze drying (figure 4-2) are transferred to the Instron-derived, stress-moisture curves for freeze-dried samples with adjusted uniform moistures and temperatures (figures 4-10 through 4-12), it is noted that for each Instron test temperature the compression during freeze drying data points fall on a straight line passing through a single moisture content as shown in figure 4-27. This means that from the Instron-derived, stress-moisture curves, a single moisture content at that particular Instron test temperature (in this case +15°C) actually simulates the compressive pressure-strain relationship (figure 4-2) obtained for a series of compression during freeze-drying experiments. To construct figure 4-27, specific compressive pressure-strain values were taken from figure 4-2 and were reconstructed on the Instron-derived, stress-moisture curve



Effect of compressive pressure on strain of green beans compressed during freeze drying

Effect of moisture content on stress to achieve given strains at +15 C (Instron tests)

Fig. 4-27. Representation of the equivalent moisture content simulating compression during freeze drying data at Instron test temperature of +15°C.

at 15°C (figure 4-11) to match the exact values of the pressures (stresses) and strains. It can be seen that by connecting the resulting points, a straight line is obtained passing through a single moisture content ($\sim$14.5%) on the Instron-derived, stress-moisture curve at +15°C which is shown at the bottom of figure 4-27. This can also be done with the Instron-derived, stress-moisture curves obtained at other temperatures as shown in figure 4-28 for the Instron test temperatures of +24°C and -10°C. However, the equivalent moisture contents that simulate compression during freeze-drying data (figure 4-2) at these other Instron test temperatures have changed values. This change in equivalent moisture content relative to the Instron test temperature is expected, and it merely reflects the fact that at a given value of the mechanical property, there are other specific combinations of moisture and temperature which result in similar compression behavior. Table 4-2 gives the specific combinations of moisture contents and temperatures, with corresponding values of the mechanical properties which simulate the compression behavior of green beans during freeze drying as a function of the applied compressive pressure (results obtained in figure 4-2). In other words, if moisture contents and temperatures of already freeze-dried green beans are adjusted to these specific combinations shown in table 4-2 or correspond to the values of the mechanical properties shown in this table, their

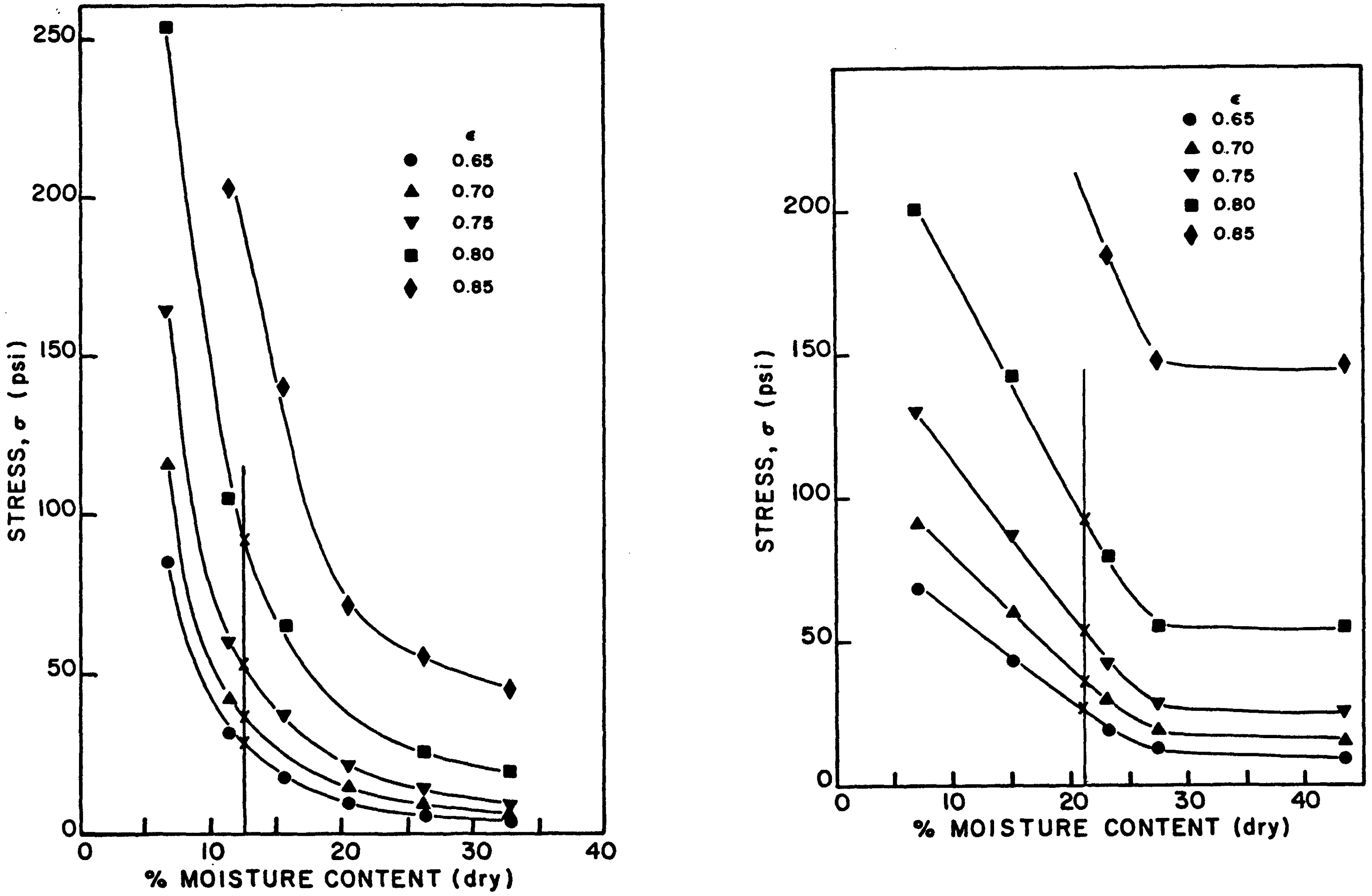


Fig. 4-28. Representation of the equivalent moisture contents simulating compression during freeze drying data at Instron test temperatures of +24°C and -10°C.

Table 4-2 Specific combinations of moisture and temperature with corresponding values of the mechanical properties which simulate compression during freeze-drying

Moisture Content % (dry basis)	Temperature °C	Tangent Modulus at $\varepsilon = 0.65$	Tangent Modulus at $\varepsilon = 0.30$	Secant Modulus at $\sigma = 21$ psi
12.7	+25°C	156 psi	30 psi	34 psi
14.3	+15°C			
21	-10°C			

stress-strain relationship as obtained in the Instron Universal Testing Machine will simulate the compressive pressure-strain data obtained in compression during freeze-drying experiments (as shown in figure 4-2).

In order to evaluate compressive pressure-strain dependency of the food in compression during freeze drying (figure 4-2), sevearl experiments have to be conducted. Each data point in figure 4-2 corresponds to one experiment in which the food is compressed during freeze drying at a constant applied compressive pressure which takes about 10-24 hours. However, rapid and simple compression tests with the Instron Universal Testing Machine ($\sim$1.5 min per test) also can determine complete compressive pressure (stress)-strain relationship of the food with a specified value of a mechanical property.

Since complete information on the values of the moduli that exist in the dry layer during freeze drying are not available, an additional compression <u>during</u> freeze-drying experiment is necessary in order to use this predictive method. Once a compressive pressure-strain data point is established for compression during freeze drying, the whole compressive pressure-strain relationship can be predicted from stress-moisture relationships which can easily be obtained from Instron studies.

The errors in compression behavior that are involved in this predictive method for ±1% deviation in the estimation of the equivalent moisture content for the Instron tests at +24°C are shown in table 4-3 and figure 4-29. It can be seen that in figure 4-29 these deviations are reasonable and within the experimental errors.

4.3.4 Evaluation of the Results Obtained in Compression during Freeze-Drying Experiments Relative to the Parameters Tested Based on the Results Obtained in Instron Studies

Using the correlations of Instron and freeze-drying studies which are presented above, the influence of various process parameters on compression behavior during freeze drying can be discussed in more depth. Table 4-4 gives a summary of the overall effects of these process variables examined in compression during freeze-drying studies.

4.3.4.1 Effect of the Applied Compressive Pressure on the Drying Rates and the Degree of Compression

As shown in table 4-4 it was noted that increasing the applied pressure acts to increase the degree of compression and to accelerate the drying process. The latter is attributed to improved thermal conductivity and heat transfer through the already dried/compressed layer. Compression process during freeze drying eliminates or decreases the void volumes between individual pieces and internal voids that are produced by sublimation of ice crystals. This results in better contact between and within the food solids and consequently a better heat transfer through the dried/compressed layer.

Table 4-3

Variations in the stresses to reach a given strain caused by ±1% variation in the estimation of the equivalent moisture content at 24°C in the Instron tests

Strain	Stress (psi)
0.65	27±3
0.70	36±5
0.75	53±7
0.80	90±15

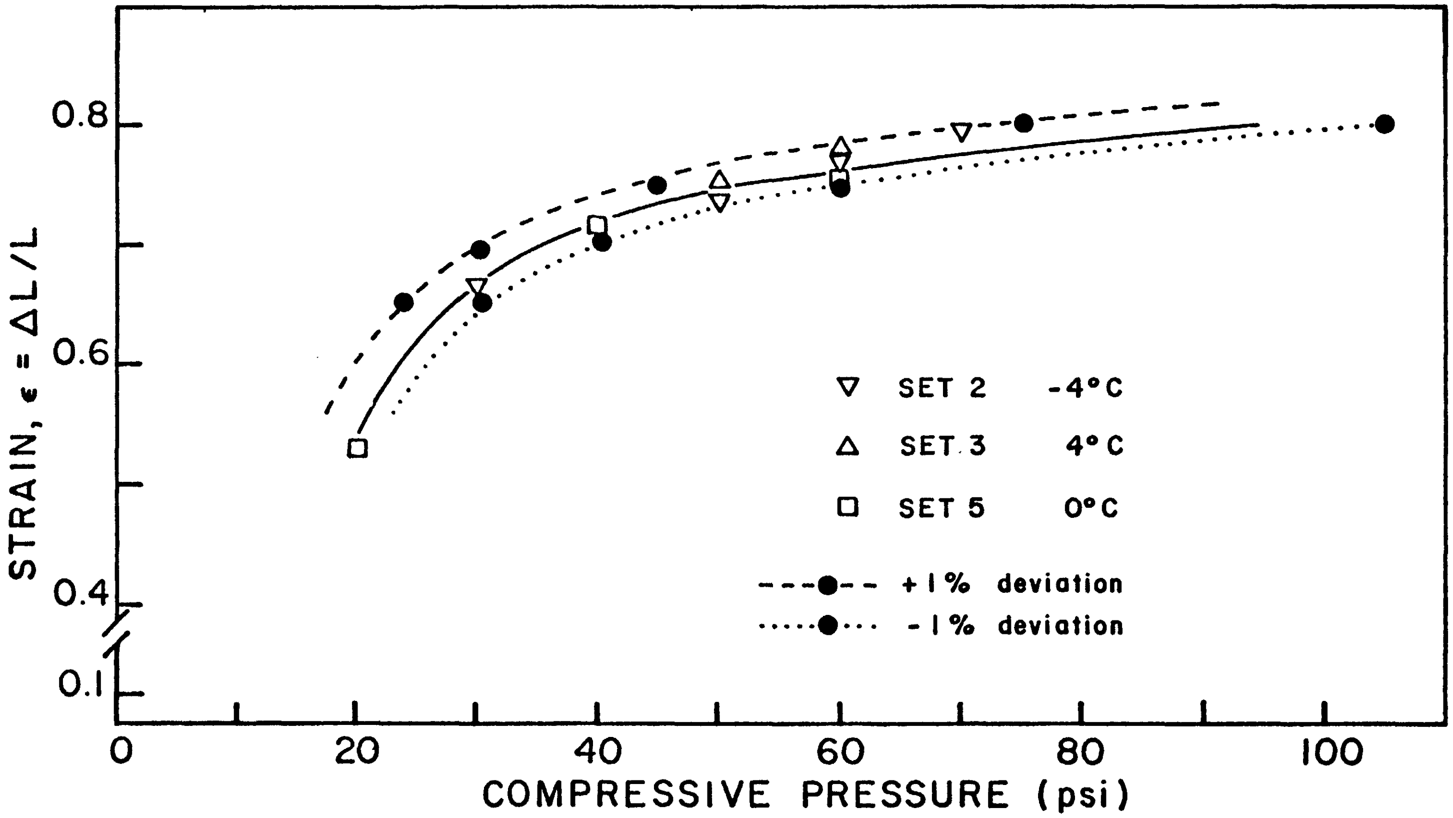


Fig. 4-29. Errors in estimation of compressive pressures to reach given strains in compression during freeze drying caused by ±1% variation in equivalent moisture content (obtained from Instron tests at +24°C).

Table 4-4 The effects of parameters tested in compression during freeze drying on final degree of compression and drying time

Freeze Drying Variable	Final Degree of Compression	Drying Time
Applied compressive pressure	Large	Some
Control temperature (heat input)	Not Significant	Large
Porosity of sample holder	Not Significant	Slight
Size of sample holder	Not Significant	None

Provided that the ice temperature remains constant, improved heat transfer results in acceleration of the ice-front movement. It was shown earlier (figure 4-26) that the rate of movement of the minimum of the modulus is a function of the rate of the ice-front movement. Thus acceleration in the ice-front movement increases the rate of movement of the minimum of the modulus. As a result of these effects, faster drying and faster and higher degrees of compression can be expected when the applied compressive pressure is increased (figure 4-6). From figure 4-6 it can be seen that these effects were observed [i.e., increasing the applied compressive pressure acted to increase both the water removal (i.e., faster drying time) and the degree of compression at given times].

4.3.4.2 Effect of Increasing Levels of Heat Input on Drying Rates and the Degree of Compression

It is clear that increasing the level of heat input accelerates the freeze-drying process. However, within the limits tested, it did not show any apparent effect on the final degrees of compression (figure 4-8). This can be explained by noting that, under the condition that the temperature of the ice region remains constant, the moisture-temperature relationship which exists in the dry layer does not change with incresaed temperature gradients across the dry layer, associated with the increased levels of heat input. This explanation is based on results obtained by Gentzler

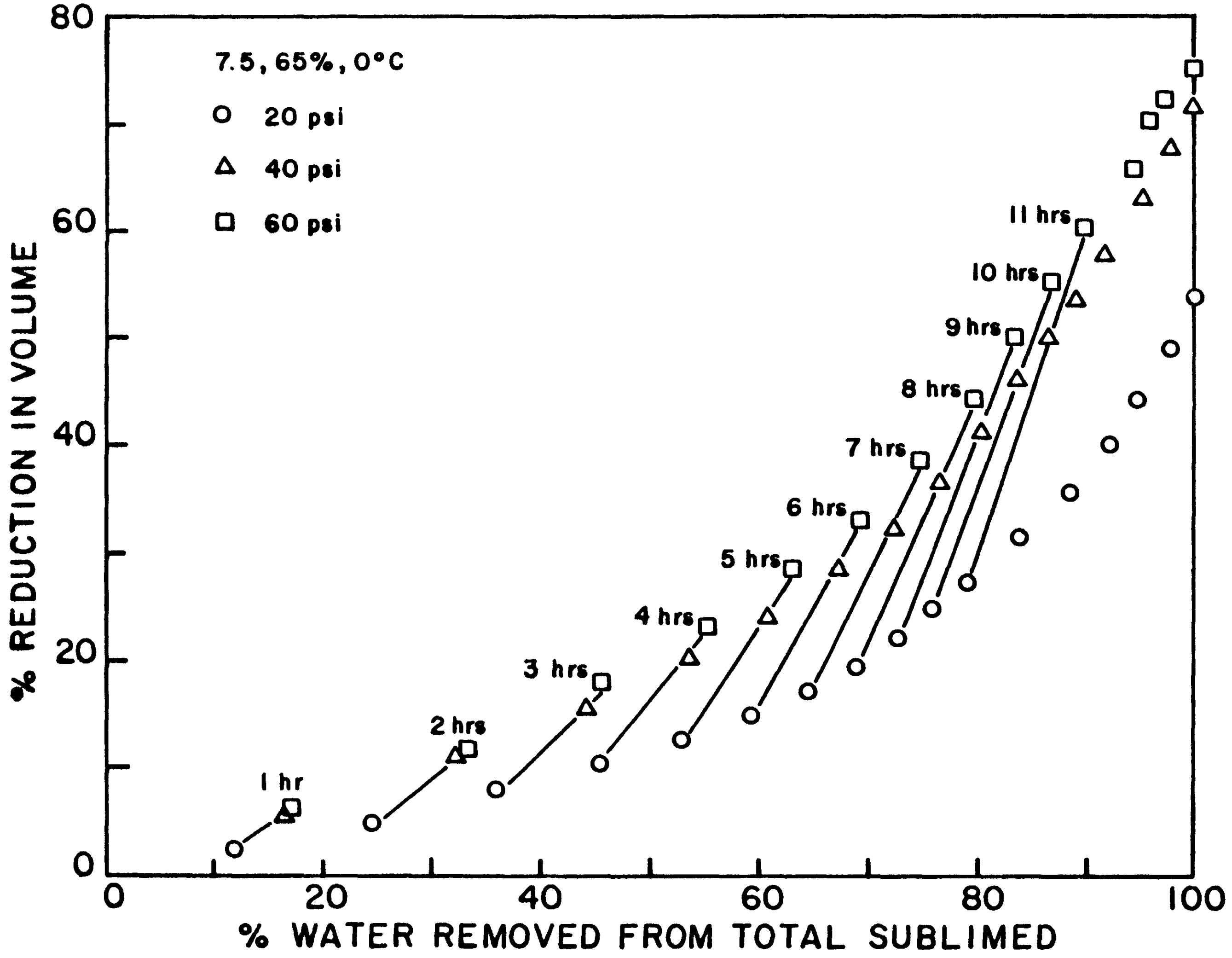


Fig. 4-6. Percent reduction in volume vs. percent water removed during freeze drying of green beans at different levels of compressive pressure.

and Schmidt (1973) which indicate that as long as the temperature of the frozen region during the freeze drying process remains constant, then the temperature and moisture contents in the dry layer zone are interrelated in such a way that at any instant in time, each temperature value in the dry layer has a unique corresponding moisture content. Furthermore, as long as the ice-front temperature remains contant, the unique temperature-moisture content relationship does not depend on location in the dry layer or time of drying. Measuring a given temperature value at a location in the "dry" layer simultaneously determines the moisture content at that location. Similarly, increased temperature gradients associated with incresased levels of heat input do not change the temperature-moisture relationship across the dry layer, provided that the frozen region temperature does not change in value. This results in the same value of the minima of the modulus (i.e., the same relative rigidity) in the dry layer, at different levels of heat input (i.e., different constant surface temperatures). This behavior is illustrated in figure 4-30. It can be seen that at a fixed ice-front temperature, the fact that the moisture-temperature relationship in the dry layer remains constant at different surface temperatures (i.e., different levels of heat input) means that the value of the minima of the modulus does not change, resulting in the same compressibility in the dry layer for a given applied

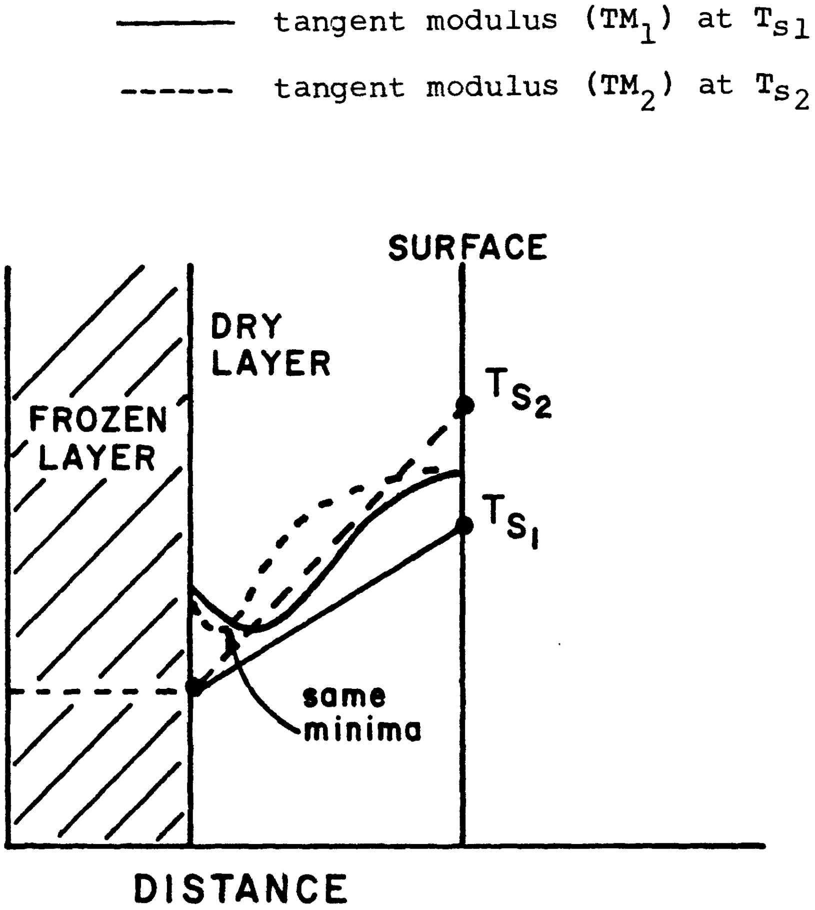


Fig. 4-30. Effect of increasing the surface temperature (T_s) during freeze drying on the range of modulus in the dry layer at constant ice-front temperature.

compressive pressure. Since increased levels of heat input accelerate the drying process and consequently the rate of the movement of the ice-front, it can be expected that the rate of the movement of the minima of the modulus will also increase. This results in a shorter process time to reach the same degree of compression and final moisture content as the level of heat input is increased (figure 4-8). As shown in figure 4-8, it can be seen that all the data points for different levels of heat inputs essentially fall on the same path (i.e., the relationship of water removal and volume reduction is the same for all heat inputs). However, at higher surface temperatures, the relative change in water removal and volume reduction is greater at any given time. For instance, as shown by the solid data points in figure 4-8, it can be seen that by the end of the 10-hour period the sample at +20°C has already reached the final drying and compression, whereas the samples at lower surface temperatures have not reached the end of the process and require more time to reach to the same values of the final moisture content and final degree of compression.

4.3.4.3 Effect of Size and Porosity of the Sample Holders on Drying and Compression Behavior

As mentioned before, the size and porosity of the sample cell holders used in these experiments did not seem to have any significant effects on drying and compression behavior of green beans, and thus there is no reason to

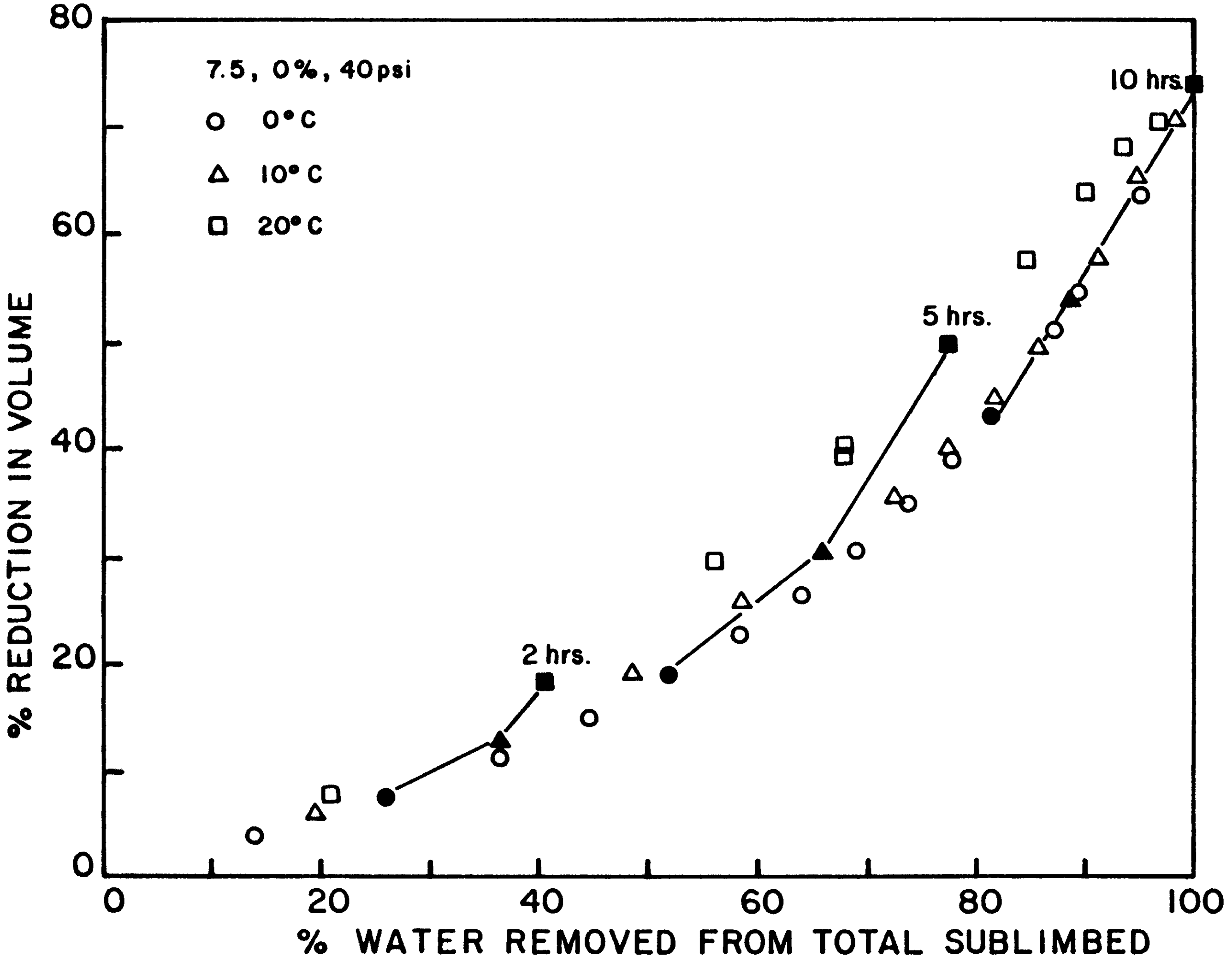


Fig. 4-8. Percent reduction in volume vs. percent water removed during freeze drying of green beans at different levels of heat input.

believe that these variables significantly alter the behavior of the modulus in the dry layer during freeze drying.

4.4 General Discussion

Compression of foods during freeze drying can be considered as an efficient method of producing high-density, low-moisture products. This technique minimizes the number of processing steps that are required to produce freeze-dried/compressed foods.

It has been shown how moisture and temperature gradients can influence the mechanical properties of the solid structure in the dry layer such that compression can be accomplished during freeze drying. Interrelationships of compression and freeze-drying processes were investigated, and the effects of some parameters that can influence compression and drying behavior of green beans were evaluated.

The independent effects of moisture and temperature on compression behavior of the food (initially freeze-dried) were investigated with an objective method (i.e., Instron Universal Testing Machine). A literature investigation showed that despite the important influence of these two variables on physical and chemical properties of food systems, there has been very little information reported in the area of rheology and mechanical properties of food porducts, especially for low-temperature and moisture levels.

The results of the above studies were further related to compression during freeze drying to demonstrate that the combination effects of the same factors are responsible for the compression behavior in both processes.

A simple method was developed in order to utilize the compression data obtained with the objective method for the prediction of the compression behavior of the foods during freeze drying.

Figure 4-26 illustrates the relationship between moisture-temperature gradients and the modulus as a function of time in the dry layer during the freeze-drying process. It can be seen that the minima of the modulus corresponds to a specific combination of moisture and temperature. Furthermore, the value of the modulus will stay constant so long as the frozen region temperature does not change irregardless of the surface temperature, or size and geometry of the sample. When there is a constant applied compressive pressure on the sample during the freeze-drying process,all the locations in the dry layer will undergo a deformation, the value of which depends on the local value of the modulus, with maximum compression taking place where the minima of the modulus is located. Considering the behavior of the minima with respect to time, it can be seen from figure 4-26 that the minima of the modulus eventually passes through all the locations in the dry layer. Thus, application of a constant compressive pressure will result in uniform deformation of the dry layer eventually resulting in a fixed degree of compression at the end of the process whose value is determined by the applied pressure and the value of the minimum of the modulus. As an example, consider the compression behavior at locations A, B, and C in the "sample" shown in figure 4-31 at various times during the freeze-drying process. As long as these locations are in the frozen region, no compression will take place as shown in the figure at time = t_0. At t_1 the ice-front has just passed the location A, so that there will be some compression at that location, the extent of which depends on the value of the modulus at location A and the constant pressure being applied to the sample. At t_2 location A has already reached its maximum degree of compression, since the minima has just passed through the location A. At time t_3 it can be seen that the value of the modulus at location A has increased, which means the relative rigidity at that location is higher than at time, t_2, and thus there will be no further compression at the given applied compressive pressure. However, at t_3 the ice-front has just passed through location B, so that some compression will start to take place at that location. At t_4 location B is compressed to its maximum degree, which is the same degree of compression as that of location A at time t_2, since the value of the minima and the applied compressive pressure have not changed. At t_5 the relative rigidity of the

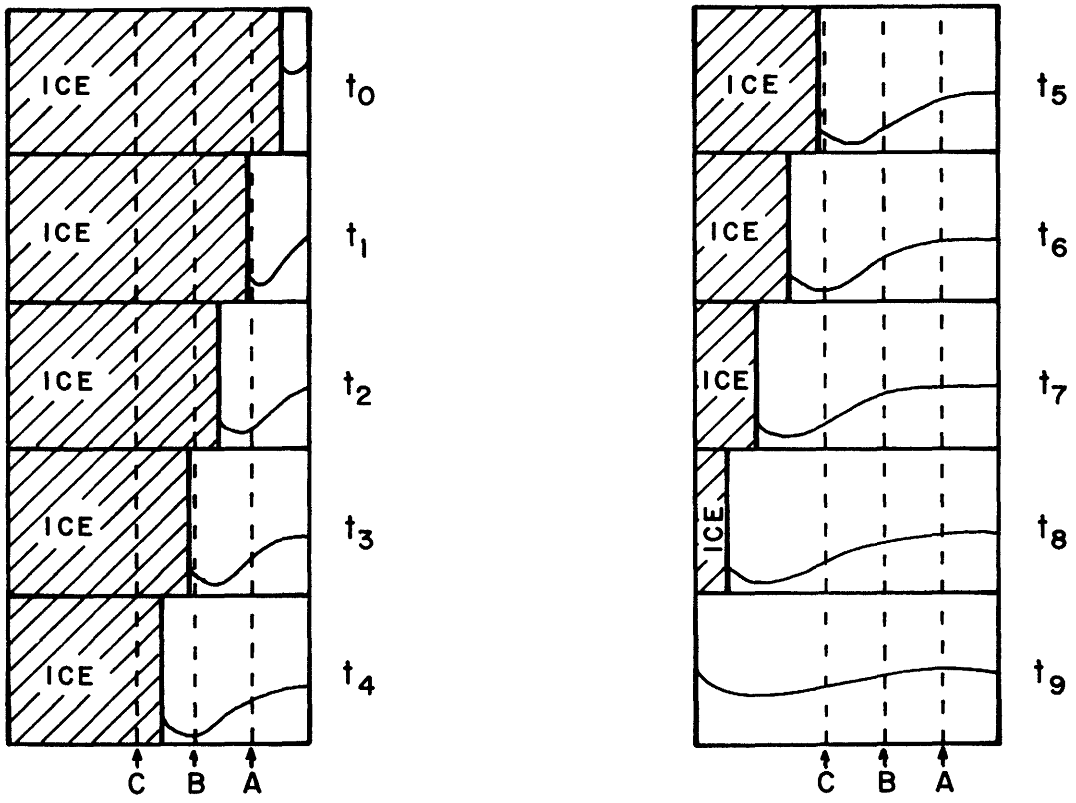


Fig. 4-31. Compressibility behavior of different locations in the dry layer during freeze drying.

location B has increased so no further compression will take place there; meanwhile (at t_5) the ice-front has just passed through location C, and compression will start to take place at this location until the maximum degree of compression is reached at time t_6. Considering that all the locations in the dry layer will eventually go through this cycle, the end result will be a uniform and fixed degree of compression throughout the sample by the end of the process. It should be noted, however, that at the very beginning and the very end of the process, compression behavior can be expected to be slightly different, due to the two end effects. Initially the drying surfaces are very cold, which results in a high value of the modulus as shown in figure 4-31 at time t_0. This results in greater rigidity of the sample surface and consequently lower degree of compression at the surface. At the end of the drying, when ice disappears and moisture gradients drop, the minima will also disappear, resulting in a rigid structure again (figure 4-31, at t_9).

The above gives the general mechanism of the compression behavior during the freeze-drying process. The knowledge of the value of the minima of the modulus and the applied compressive pressure will give information on the final degree of compression. Since there is not enough information on the values of the mechanical properties of the dry layer during freeze drying to theoretically calculate compression behavior, the simple predictive method which was explained in detail in section 4.3.3 can be used to predict the compression behavior of foods during freeze drying.

5. CONCLUSIONS

The results and conclusions of this study may be summarized as follows:

A. Study of mechanical properties (Instron studies)

1. Increases in moisture content above the B.E.T. monolayer value and up to about 25% (dry solid basis) decrease the rigidity of freeze-dried green beans which have been rehumidified.

2. The freeze-dried product, rehumidified up to the moisture content of approximately 25% can be compressed with no apparent structural damage. The compressed product partially recovers upon unloading and has high recovery upon rehydration.

3. Increasing temperature from -25°C to +24°C in the freeze-dired product having moisture contents of approximately 6%-30% also reduces the relative rigidity. However, the effect of temperature is less significant than that of moisture.

4. For given values of the mechanical properties there are specific combinations of moisture and temperature which result in equivalent compression behavior (i.e., similar stress-strain relationship). Therefore, the compressive behavior can be predicted at any given combination of moisture and temperature.

5. Based on the information in the literature it can be deduced that for each temperature in the dry layer during freeze drying there is a unique moisture content, and for each combination of temperature and moisture there is a corresponding value of each mechanical property. On this basis, by consideration of the moisture and temperature gradients that exist in the dry layer during freeze drying, it can be stated that at each instant in time, each location in the dry layer will have a different mechanical property (i.e., different compressibility characteristics). This results in a range of mechanical properties throughout the dry layer which change value at each location as the ice-front recedes with time. Furthermore, as long as the temperature of the ice-front remains constant, the value of the minimum modulus will not change.

6. It can be expected that application of compressive pressure at any instant in time during freeze drying will cause compression of the dry layer. The degree of compression, however, at each location will depend on the stress-strain relationship at that location. Furthermore, the maximum compression will take place at locations which have lowest values of the modulus (minima).

7. Considering that the fixed value of the minimum modulus (rigidity) will eventually pass through all locations in the dry layer during freeze drying, at a constant applied compressive pressure, all locations in the dry

layer will eventually be compressed to the same ultimate degree.

B. According to the results obtained in the compression during freeze drying experiments and part A, it can be stated that:

1. The main process parameter that affects the degree of compression during freeze drying is the level of the applied compressive pressure.

2. Application of compressive pressure during freeze drying also increases drying rates. This is presumably due to improving heat transfer (and in particular the thermal conductivity) through the already dried/compressed layer.

3. Higher heat inputs to accelerate the freeze drying process result in faster movement of the minimum modulus and, therefore, in faster compression. However, when the temperature of the ice-front remains the same, similar final degrees of compression will be obtained for a given compressive pressure, at different heat inputs.

4. Size and porosity of the sample holders (used in this study) which do not change the ice-front temperature will not affect the minimum modulus; thus, similar degrees of compression will be obtained at the given applied compressive pressure.

6. FUTURE RESEARCH

The research reported upon here has described and explained the compression behavior of green beans, both under iso-moisture/iso-thermal conditions (Instron studies) and under freeze drying.

A more complete and wide-ranging understanding of compression behavior of foods at low moistures and/or temperatures and during freeze drying can be achieved by the future research in the following areas.

1. The independent and interactive effects of moisture and temperature on mechanical properties of other freeze-dried food materials using the Instron Universal Testing Machine.

This area of research relates to the variation of the basic material structure and its effects on compression behavior. It can be expected that the composition and organization of the structural polymers (such as carbohydrate-based polymers in plants and protein-based polymers in animal tissue), and those low-molecular-weight compounds that are located between the polymeric segments result in different mechanical properties which can be affected differently by moisture and temperature. It is thus important to investigate the influences of moisture and temperature on compression behavior of other foods to demonstrate the existence of specific combinations of these two variables which result in common values for mechanical properties and in a similar compression response of the given food (irregardless of the basic material structure) under stress.

2. To investigate the effects of moisture and temperature on mechanical properties of a model system or artificial food structures. The advantage of using a model system simulating foods is that it is easy to achieve major changes of the composition in order to evaluate the effects of the basic structural components in the model system.

3. A more detailed study of the conditions which result in collapse and irreversible structural changes due to compression. Although some tests were conducted to evaluate the effect of the level of heat input on collapse, further studies are required to collect sufficient information in order to be able to predict the conditions which result in irreversible structural changes in compression during freeze drying.

4. Evaluation of the microstructural changes that occur due to compression at different levels of moisture content and temperature and various basic material structure, using microscopic techniques. Rahman et al. (1969) reported that the most damage to the cellular structure of freeze-dried/compressed carrots was due to the freezing process prior to dehydration and compression and further that compression

of the product caused no significant damage to the cellular structure. However, further work on microstructural changes due to compression would be of interest especially with respect to collapsed systems. This area of research relates to the evaluation of the microstructural changes that occur at various conditions which result in collapse and irreversible structural changes in compression of food materials.

5. Determination of moisture and temperature gradients that exist in the dry layer of various foods during freeze drying (espeically moisture levels close to ice-front) at different conditions of heat and mass transfer. This will give the knowledge of the mechanical properties and thus compressibility of the dry layer using the data obtained for the same foods from the Instron studies. This would allow prediction of the compression behavior during freeze drying.

6. To model a mathematical simulation of freeze drying which includes changes in food physical properties due to compression. It should be realized that due to the complex nature of the process, a number of simplifying assumptions will probably have to be made. The mathematical model can be used to predict the time dependence and overall compression with freeze drying by incorporating the compression information obtained from the Instron studies

directly into the model. This will eliminate the need for actual freeze drying experiments for the prediction procedure. The overall methodology will follow directly from those used in the mathematical simulation, an iterative procedure over small time intervals in which heat and/or mass transfer balances would be used to evaluate temperature and moisture gradients, water loss, and interface movement. The mechanical properties for each time interval, which are a function of moisture and temperature at each location, will be obtained from the information obtained with Instron tests. The values of the mechanical properties would be combined with the value of the applied force to calculate the extent of compression at each location. Changes in food physical properties due to the compression would be made prior to the next iteration.

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