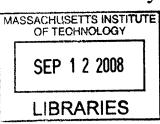
Potential Applications of a Toughened Silicon-Based Alloy

by

Wang S. Lei



SUBMITTED TO THE DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF ENGINEERING IN MATERIALS SCIENCE AND ENGINEERING AT THE MASSACHUSETTS INSTITUTE OF TECHNOLOGY

SEPTEMBER 2008

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Abstract

Silicon has long been used as an alloying element in various metal alloys, in engineered ceramics, and in the semiconductor industry. However, due to its intrinsic low fracture toughness, it is generally perceived as a poor choice of material for mechanical applications. This study explores some potential short and long term applications for a new type of castable silicon-rich alloy with an increased fracture toughness, by utilizing several different material selection indices.

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Introduction

Current Uses of Silicon

Today, roughly 5.1 million metric tons of silicon metal is produced in the world each year, with China being the leading producer at 2.9 million metric tons. In comparison, the US produces about 150,000 metric tons per year [1]. Currently, the biggest uses for silicon are as an alloying element in aluminum and ferrous alloys, as a major component in glass and concrete, and in silicone production. Perhaps with the most importance, but at much a much smaller consumption percentage, silicon is the most widely used element in the semiconductor industry.

The Low Fracture Toughness of Silicon

Despite having some desirable mechanical properties such as an intrinsic high strength and hardness, silicon has not been considered in many mechanical applications mainly due to its low fracture toughness (<1 MPa* m^{1/2}), which is even more brittle than glass. In an effort to look for new ways to utilize this material, a castable, silicon-rich (>50 weight %) alloy is under development at MIT to increase silicon's fracture toughness to the point where it can be considered for mechanical applications. The research is still in early stages, but has shown great promise that the fracture toughness of silicon can be raised between two to ten times its original value.

Much like a ceramic, brittle fracture is the main mode of failure of silicon at room temperature. This is mainly due to the strong covalent nature of elemental silicon bonds, which makes dislocations extremely difficult to form and propagate, rendering fracture a more likely mechanism of accommodating applied displacements. This means that silicon does not undergo any plastic deformation before failure. As seen in Fig. 1, large grains (on the order of millimeters) dominate the microstructure of cast polycrystalline silicon. Without many grain boundaries to act as deterrents, small cracks can propagate through the grains quickly and largely unimpeded, leading to fracture. This is especially true when silicon is loaded in tension. The first step in commercializing a silicon-rich alloy product must be to address the problem of crack formation and propagation through silicon's microstructure.

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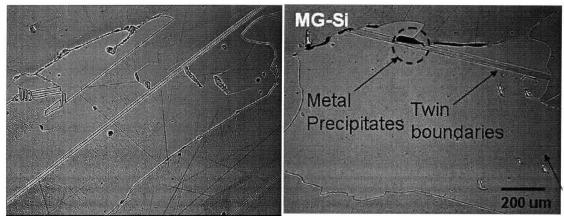


Fig. 1. Light microscope images of the large grains in metallurgical grade silicon [2]. Metal precipitates and twin boundaries are examples of some of the infrequent obstacles a crack may encounter in pure silicon

Microstructural Engineering

Understanding the need to deflect and stop crack propagation, the Schuh group has begun looking into the strategy of microstructural engineering to address the problems of crack propagation and extremely low fracture toughness in silicon, using a common metallurgical practice to alloy a brittle material with secondary elements and phases in order to increase fracture toughness. By alloying with metallic elements at near-eutectic compositions, the alloying elements can achieve predictable uniform homogeneous microstructures within the host element. There are currently two ways to achieve toughening effects with different alloying elements.

The first is through the inclusion of second phases that can interact with cracks. Many elements form homogenous second phases when added to silicon, and form throughout the microstructure when alloyed in a melt and cast. The presence of the second phase aids in deflecting cracks through interface interactions with the pure silicon phase. In the second mode of toughening, a terminal ductile phase results when the alloying element is added to the composition. This secondary phase acts as an obstacle to cracks initiated in silicon-rich phases of the alloy, allowing energy to be dissipated through plasticity within the ductile phase.

Current research is ongoing in finding the right combination of alloying elements and compositions that would maximize the effects of alloying to increase silicon's fracture toughness.

Engineering Ceramics

Engineering ceramics, like zirconia and alumina, have long been utilized in high temperature and high strength applications that simply can't be addressed by metals. As early as the 1960s, ceramics have been under research to replace metal alloys as the main materials used in turbine components design due to the promise of increased efficiency from the higher turbine inlet temperature [5]. However, the promise of ultra efficient turbines has not been fulfilled at the time of writing due to the inconsistent operation hours to failure of most prototypes [6]. This is caused, in part, by the low fracture toughness of ceramics, which is much lower than metals but at a level the proposed silicon alloy can realistically attain $(1-15 \text{ MPa}* \text{ m}^{1/2})$.

While the new castable silicon alloy may or may not reach the same performance level as some engineering ceramics, it will definitely compete in terms of cost. The difference lies in the cost of production, as most ceramics employ some type of powder sintering method of production, while the proposed alloy will use much cheaper casting methods.

Competing Intellectual Property

A look at the intellectual property landscape of new and unique alloys shows very little in the realm of Si-rich alloys intended for mechanical applications. This is not surprising considering the well-established roles silicon has filled in the last 50-plus years mainly as an alloying element and in the semiconductor industry.

United States Patent #5,833,772

This is the patent most relevant to the current project, filed by Elkem ASA entitled Silicon Alloy, Method for Producing the Alloy and Method for Production of Consolidated Products From Silicon. Dated Nov. 10, 1998, this patent claims a wide range of silicon alloy compositions produced through "rapidly solidifying" a melt of the

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following composition: 2-40% by weight Al, 15-45% by weight Ti, 0-10% of at least one element selected from the group consisting of V, Cr, Mn, Fe, Ni, and Co, and at least about 35% by weight Si [7].

This is an important patent because it provides a claim involving numerous potential alloying materials that might be used to engineer the structure of a silicon alloy. It would be crucial for the final composition of the alloy not to encroach on the range covered by this patent. An encouraging note to take away from this patent is that nothing concerning the alloy's fracture toughness is mentioned in the patent. It stands to reason that the authors' main area of concern is with the strength of the alloy, as that is mentioned throughout the patent.

United States Patent Application Publication #US 2006/0013721

This is another patent applied by the same Norwegian company Elkem ASA, entitled *High Strength, Oxidation and Wear Resistant Titanium-Silicon Base Alloys and the Use Thereof.* Dated Jan. 19, 2006, this application attempts to claim an alloy with the following composition: 2.5-12% by weight Si, 0-5% by weight A1, 0-0.5% by weight B, 0-2% by weight Cr, and the remainder Ti. The patent application lists several potential applications for this alloy, including "connecting rods, piston crowns, piston pins, inlet and outlet valves" in engines, "surgical implants, bone plates, joints" in the medical field, and "static blades, in axial flow compressors and fan blades in jet engines" [8].

This patent application appears to be unrelated to patent #5,833,772 based on its range of claimed compositions. What is of small concern in this application is the mention of "fracture toughness of more than $K_{1c} = 15$ MPa* m^{1/2}" towards the end of the application as part of its claims, indicating Elkem ASA's desire to produce silicon related alloys with enhanced fracture toughness. However, this application is mainly a Ti based alloy, not a Si alloy, so should not encroach on the proposed alloys.

Favorable Design Indices of Silicon

With the work of increasing fracture toughness of silicon well under way, it is now important to look at where this potentially toughened silicon-based alloy can excel in application. The material selection software developed by Granta, CES Selector, was used to compare mechanical, thermal, and other material properties and parameters of silicon against other structural materials with similar properties. There are a multitude of possible ways to create an "Ashby plot" using different combinations of a material's properties, so to effectively create one that is useful, we must consider the beneficial aspects of the material in question. In this case, silicon has a low density, low cost, high Young's modulus, high compressive strength, good thermal conductivity, and low thermal expansion coefficient. Using these properties as guidelines and constraints, several favorable design indices were plotted using the CES Selector, suggesting some potential applications. Throughout the following sections, different materials classes will be represented using the same color scheme, which is laid out in Figure 2.

Price Related Indices

Some of the following plots deal with prices of materials. For the most part, the CES Selector provides a reasonable price range for the materials used in the material selection index plots. However, in the case of Silicon, the price range that was provided by the software was 9.124 - 12.14 per kg. These are likely prices for solar or semiconductor grade Silicon, being much higher than the trading price listed by the US Geological Survey of 1.50 per pound (3.3 per kg) for metallurgical grade Si [14]. Considering that new structural silicon alloys will be based on metallurgical grade silicon, it is reasonable to argue that the USGS price should be used in any material selection index. This has been done in the plots that follow, where the CES price-based results are also shown for comparison in hatch-marked areas.





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Mechanical Properties

To show both the major strength and weakness in the same space, Fig. 3 is a plot of price normalized compressive strength vs. price normalized fracture toughness. While it has very high compressive strength, silicon is about an order of magnitude away from competing with engineering ceramics and ferrous alloys even at its corrected normalized price. An order of magnitude increase is ambitious at this stage, but may be possible given significant progress in microstructure design.

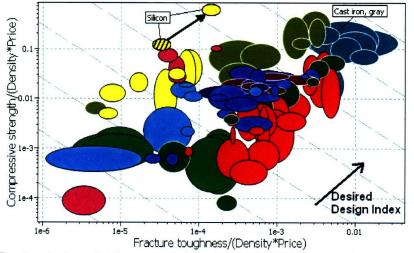


Fig. 3. Design index plot for materials suitable for high strength and toughness applications.

The first design index we looked at was for materials suitable for cutting purposes. In order for a material to perform well in cutting, it must have high compressive strength. The constraint on the load at the point of contact of an edge is proportional to:

$$\left[\frac{PE^2}{R^2}\right]^{1/3} \le A\sigma_c \tag{1}$$

where *P* is the load, *E* is the Young's Modulus, *R* is the radius of curvature of the material at the edge, σ_c is the compressive strength, and *A* is a constant. Rearranging the above term yields the following term for the maximum load that a material is able to bear on an edge as:

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$$P_{\rm max} = A^3 R^2 \left[\frac{\sigma_c^3}{E^2} \right]$$

The material selection index in this case would then be:

$$C = \left[\frac{\sigma_c^{3}}{E^2}\right]$$
(3)

(2)

It is interesting to note that according to this index, materials with a high modulus will appear to be a poor candidate in this application. Fig. 4 shows the plot of this selection index in log scale, with guidelines of slope 3/2, with the desirable candidates skewing towards the upper left region. Strong candidates include thermosets and elastomers, which are already used for plastic cutleries. However, silicon is a strong candidate for this application as it can take on a much higher load than thermosets and elastomers, which means it can compete with metal cutleries should the fracture toughness be increased.

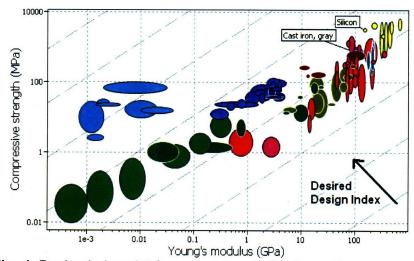


Fig. 4. Design index plot for materials suitable for cutting purposes.

The abrasive wear parameter is a design parameter used by ceramics manufacturers to determine the effectiveness of materials to withstand an abrasive environment [16]. Because we are trying to gauge the new alloy's performance with that of engineering ceramics, this parameter could be used to find a relevant design index, and is given by:

$$A = K_{1c}^{.5} H_V^{1.43} E^{-.8}$$
(4)

where K_{1c} is the fracture toughness, H_V is the hardness, and E is the Young's Modulus. Combining this term with the per-volume price yields the following design index:

$$C = \frac{K_{1c}^{5} H_{V}^{1.43} E^{-.8}}{p\rho}$$
(5)

In Fig. 5, the abrasive wear parameter is plotted against per volume price of various materials, with guidelines of slope 1 denoting indifference between price and performance. From a purely performance-based standpoint, silicon is already competitive with most metals, and can only improve with the increased toughness of the new alloy. When price is factored into the material selection process, it can be seen that, at the corrected price, silicon would be chosen over all engineering ceramics as an abrasion resistant material. However, it is clear that with a significant increase in toughness, silicon stands to compete against virtually every other material class as an abrasion resistant material.

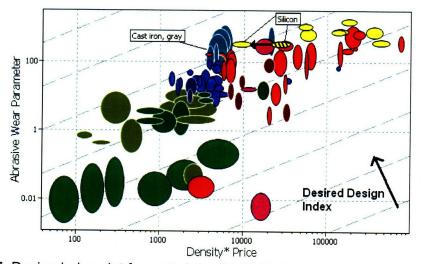


Fig. 5. Design index plot for materials with high tolerance for abrasive wear.

Thermal Properties

Silicon also has very good thermal properties. It is a very good thermal conductor, has a high specific heat, and a low thermal expansion coefficient. Based on these properties, the following material selection indices were found to be favorable to a potentially toughened silicon alloy.

The first heat-related material selection index is for materials that minimize thermal distortion. Materials that are resistant to thermal distortions are ones often chosen for precision temperature devices, so often experience steep temperature gradients within its bulk and immediate surroundings. The heat change within a material is given by Fourier's law as:

$$q = -\lambda \frac{dT}{dx} \tag{6}$$

Where q is the total amount of heat added to the system (in this case, the bulk of the material), λ is the thermal conductivity, and $\frac{dT}{dx}$ is the temperature gradient in the material. The term for thermal strain is given as:

$$\varepsilon = \alpha (T_0 - T) \tag{7}$$

Where ε is the strain, α is the thermal expansion coefficient, and $T_0 - T$ gives the temperature difference between the material and ambient environment. Combining equations 6 and 7 yield the following term for thermal distortion caused by a temperature gradient in a system as:

$$\frac{d\varepsilon}{dx} = \frac{\alpha dT}{dx} = \left[\frac{\alpha}{\lambda}\right]q$$
(8)

This shows that by maximizing the following material selection index, thermal distortion would be minimized:

$$C = \frac{\lambda}{\alpha} \tag{9}$$

At roughly 140-150 W/m*K, it is a better conductor of heat than cast iron and other ferrous alloys. This means that silicon has a great ability to distribute heat quickly and evenly throughout. Couple this with a low thermal expansion coefficient at 2.2 μ strain/°C, this makes silicon a great material for minimizing thermal distortion. Fig. 6 shows a plot of the design index for minimizing thermal distortion, with guidelines of slope 1 as the indifference point between thermal conductivity and thermal expansion coefficient. Silicon performs in this application better than almost all currently available materials. While no tests have been done to test the new alloy's thermal conductivity or

expansion coefficient, the alloy is expected to have a slightly lower design index than pure silicon but should still be superior to most competing materials.

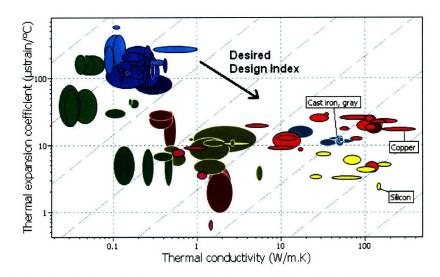
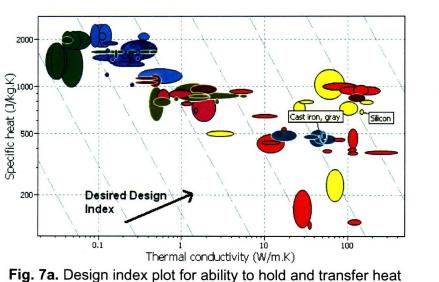


Fig. 6. Design index plot for materials that exhibit minimal thermal distortion

Fig. 7a shows the second plot that is of interest: specific heat vs. thermal conductivity. This design index is again taken from ceramics manufacturers that want to determine a material's ability to hold and transfer heat effectively, given as:

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$$C = C_V \alpha \tag{10}$$

where C_{ν} is the specific heat and α is the thermal conductivity. Again, silicon, with a specific heat of ~700 J/kg*K, appears to perform much better than ferrous alloys.

However, the above index does not take into account the density of materials. The more useful index would be one where the material's ability to hold and transfer heat evenly is based on the volume of the material present, and is given by:

$$C = C_{\nu} \alpha \rho \tag{11}$$

The amount of heat held by silicon per volume is lower than cast iron, as shown in Fig. 7b, which could be a problem in a heat storage and distribution application.

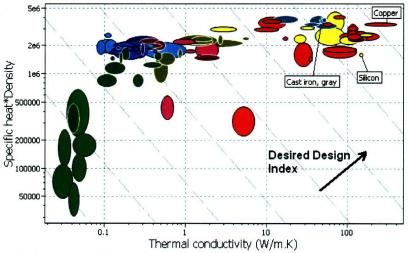


Fig. 7b. Design index plot for ability to hold and transfer heat based on the material's relative density

When dealing with heat-related applications, it is often important to have materials that are resistant to thermal shock, as a gradient in temperature between the surroundings and the material is usually inevitable. To determine the amount of thermal shock a material can withstand, one must look at the thermal stress the material experiences when there is a change in temperature, given by:

$$\sigma = \frac{E\alpha}{(1-\nu)}dT\tag{12}$$

where ν is the Poisson's Ratio and σ is the thermal stress the material for a change in temperature, dT. Assuming that the material experiences brittle fracture, it will occur in the material if this thermal stress exceeds the tensile strength of the material, so:

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$$\sigma = \sigma_t \tag{13}$$

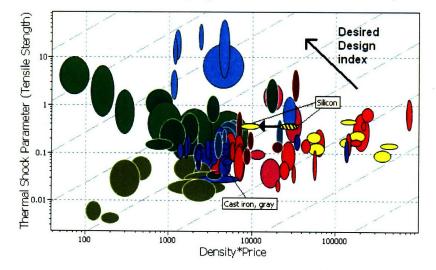
Rearranging the terms shows that the largest change of temperature a material can withstand is given as:

$$\Delta T = \frac{\sigma_t (1 - \nu)}{E\alpha} \tag{14}$$

Materials that maximize this range of temperature would be able to withstand the largest thermal shocks. So the best materials to be chosen to minimize thermal shock at a given per-volume price would be one which maximizes this material selection index:

$$C = \frac{\sigma_t (1 - \nu)}{E \alpha p \rho} \tag{15}$$

Silicon is relatively robust against thermal shock. Fig. 8 shows the thermal shock parameter vs. per volume price of various materials, calculated using tensile and compressive strengths, respectively. Even in the case where the tensile strength of silicon was used, it still is more resistant to thermal shock than cast iron and all other engineering ceramics other than silicon nitride. It is only surpassed in ability to withstand thermal shock by elastomers, which have much lower melting points, so would not be able to be used in high temperature applications. For a properly toughened alloy, the strength in tension and compression will be similar, and close to the compressive strength of silicon. In that case, silicon outperforms all ceramics and metals in terms of thermal shock resistance; only organic materials and a few foams offer competition on this basis, and these would generally be soft and compliant. Thus, silicon has a unique advantage in terms of its combination of mechanical properties and thermal shock resistance.



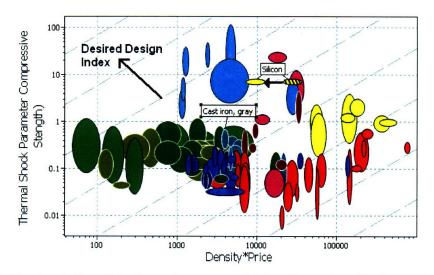


Fig. 8. Design index plots for various materials' ability to handle thermal shock calculated using their compressive and tensile strengths.

Discussion

Possible Applications

After looking at various design indices, it is clear that potentially toughened silicon can be great for cutting tools and heat related appliances, such as cookware. Toughened silicon can compete well with currently available stainless steel knives based on cost and density – essentially a knife that is lighter and costs less. In cookware, the toughened silicon should be able to compete with cast iron skillets and other metal-based pots and pans due to its extremely small thermal distortion and ability to withstand thermal shock. It may not necessarily be cheaper than current cookware, but it could win out if it proves to deliver higher performance and endurance. Due to the highly segmented nature of the markets of these applications, a more in-depth analysis of potential market penetration would represent a significant effort, beyond the scope of this thesis.

One potential application for toughened silicon that was given a more thorough analysis in this study was as a building material for microturbine parts.

Introduction to Microturbines

A microturbine is a small combustion turbine which employs the Brayton Cycle to generate anywhere from 25 to 500 kW of power at 20-30% efficiency. However, since most microturbines are configured for Combined Cooling and Heating Power (CCHP),

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most units have an overall efficiency of over 80% – a huge selling point for microturbines. However, even with these promising numbers, the installation cost for microturbines still put them behind the traditional power grid system in terms of dollars per kW generated.

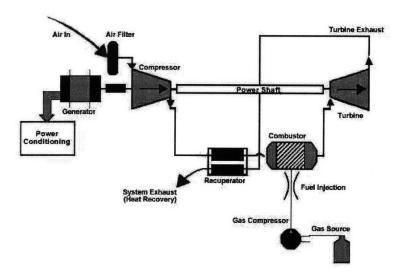


Fig. 9. A diagram depicting the power generation process of a microturbine [9].

The Microturbine Market

Microturbines remain in a niche market as a source of electricity. Today, microturbines make up roughly 11% of all gas turbine production [10]. The market is dominated by Capstone Turbines, which has captured more than 80% of the market share. Other players include Ingersoll-Rand, Solar Turbines, GE, and UTC. Together, it is estimated that the industry has overall annual revenue of roughly \$25 million [11].

There was a significant amount of hype involved with DGS at the time of the energy crisis in California. When the microturbine market leader, Capstone Turbines, went public on June 29, 2000, the initial public offering was priced at \$27.37 per share. This is also the same time when the Department of Energy began a 6-year Advanced Microturbine Systems Program Plan [12]. This plan was a cooperative agreement by Capstone, GE, Ingersoll-Rand, Solar Turbines, and UTC to improve on the next generation of microturbines. In less than three months, buoyed by speculations that this

technology would become the new way of energy production and in the midst of the California crisis, Capstone's stock hit a high of \$98.50 per share.

However, the market demand for microturbines never approached the expectations of analysts – approximately 5000 units have been sold in the whole industry in 9 years since the first unit was sold by Capstone in 1998 [10] – due mostly to the lack of real improvement in technology. Among other objectives, the Department of Energy asked the companies to improve efficiency to 40% or better, reduce NO_x emission to less than 7 ppm, and the reduce power cost to below \$500 per kW-hr [12]. A lot of these objectives were not met or were recognized to be too difficult to achieve. Soon, the stock price soon plummeted, and today, it hovers around \$3.32 per share.

Lessons learned

While the Advanced Microturbine Systems program did not lead to many substantial improvements other than a recent low emission microturbine model from Capstone, it did show that major improvements in material properties are essential for the future designs of microturbines. Engineering ceramics, especially silicon carbide and silicon nitride, have long been looked at as potential materials in turbine development, and the results of the program's findings paint a fairly clear picture of material properties that would be needed for a successful ceramic turbine material candidate, the most salient of which are [12]:

- High Strength: >1000 MPa
- High toughness: >10 MPa*m^{1/2}
- High Weibull modulus (to ensure uniformity): >20
- Long operating life: >20,000 hour field testing

Effect of New Alloy on the Microturbine Industry

The microturbine industry is at an interesting place. It has a product which is very promising and has positive signs, such as the encouraging government policies and research allowances in the field, pointing to major adoption in the market. But it is simply not efficient enough to do so at this time. It is an industry where the application has been fairly well-defined, but the research side has not caught up to the needs of the market. However, there is definitely still expectation that this industry can rally. This can be gleaned from the steady rise of the stock price of Capstone Turbines. The introduction of a new silicon-based alloy offers a weight and cost benefit over both metals and engineering ceramics, potentially be able to provide a turbine inlet temperature of over 1300 °C, both of which would increase the efficiency of the microturbine system.



Fig. 10. Stock price of Capstone Turbines (CPST) over the past year. Notice the steady upward trend following the crash in 2000 [13].

Cost Model

Because the new alloy is in such an early stage that neither the desired alloying element(s) nor method has been finalized yet, it is hard to provide a concrete cost model to follow in developing it. However, we do know that the current microturbine production cost is \$700-\$1100 per kW, while the traditional production cost of the power grid system is around \$400 per kW [15]. This means microturbines could conceivably compete with the grid system if their efficiency was improved by ~28%.

If we now compare the cost of material between the base material and nickel – an essential material in metallic turbine parts – a very rudimentary guideline as to how much the new casting method should cost could be established. Silicon is more than 10 times cheaper than nickel, and is more than 3 times less dense. As a bounding analysis, the cost

to produce the alloy can not be more than 30 times more expensive than the base element if it hopes to even compete with current microturbine technologies.

Recommendation

From just a cursory look at one of the possible applications for the proposed new alloy, one can see that there is a clear demand for such an alloy. The interesting aspect of this project is how developed the applications already are, while the research side is still very underdeveloped. I believe that the knowledge of where the technology will need to be will help steer the alloying process towards the right path in this long term project. As such, I think research on this new type of alloy would be best in a university setting, where there is less risk involved when working on a long term project, and the researchers can have more time dedicated to improving the properties of the base metal.

When the desired alloy is finally produced, I believe the best strategy would be to protect the composition and possibly the alloying procedure with patents. It would be a mistake to try to approach the IP landscape from the application side, as microturbine companies have long held the IP to many DGS technologies. Instead, it would be advisable to enter the industry as the proprietary provider of this new type of silicon based alloy.

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