Experiments and Design Considerations toward a Novel Continuous Flow Aluminum Fuel Reactor for Underwater Vehicles

by

Zachary S Rolfness

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Abstract

Presented here is experiments and design consideration toward the development of an activated bulk aluminum slurry and liquid water reactor. Previous work has shown successful activation of bulk aluminum for 99% successful oxidation with water. However, this reaction has only been seen in bulk reactors with only a bit of progress toward slurry reactors

Several experiments were conducted over the semester. The results for high-pressure activated aluminum showed a positive correlation between initial pressure and reactivity. This trend held for both experimental results for the BB form and the slurry form. Experiments on the development of a homogenous slurry showed that a higher shear rate mixer and double the amount of fumed silica were needed to produce a homogenous non-clumping aluminum slurry. Lastly, pumping experiments, unfortunately, showed that in its current state, the fuel cannot be reliably pumped due to the settling of the aluminum inside the piping. Solutions are discussed to overcome the issues seen while pumping.

Among other contributions, a theoretical reactor design is presented that hopes to eliminate issues with jamming seen during experimentations. Future hope is that with the contributions found in this paper, further work can be done to successfully engineer a continuous flow aluminum slurry reactor for a wide array of applications.

Thesis Supervisor: Douglas P Hart Title: Professor of Mechanical Engineering

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Introduction

Aluminum is the most commonly found metal in the Earth's crust. Although it is often used for structural and packaging applications, scientists and engineers have long sought a way to tap into its immense energy potential. Aluminum has one of the highest energy densities of any non-nuclear material, with more than twice the volumetric energy density of gasoline or diesel fuel, however not until the last 10 years has that energy potential been able to be realized. Naturally, a stabile oxide layer forms around the surface of aluminum when in an oxygen-rich environment which makes it difficult to extract energy from aluminum.

In the last decade, researchers at MIT were able to develop a novel way to access this energy found inside aluminum, by activating it using a gallium and indium eutectic. This resulted in the creation of a new, energy-dense solution to store both hydrogen and energy within aluminum pellets. The engineering applications are vast, especially with a push for cleaner and safer energy storage solutions. Aluminum energy storage has none of the safety and environmental concerns that come with lithium-based energy storage systems, and none of the losses associated with other potential energy storage systems.



Aluminum as a Hydrogen and Heat Source

Figure 1 Materials plotted by gravimetric energy density (MJ/kg) vs volumetric energy density (MJ/L)

Aluminum when reacted with water is one of the most energy dense materials of any non-nuclear material (at approximately 84MJ/L, which is more than twice the energy density of gasoline (34 MJ/L) or diesel fuel (39MJ/L). When reacted, activated aluminum produces heat (15.5KJ/g-Al) and hydrogen gas (0.11 g-H2/g-Al). The heat energy can be used for processes or mechanical work extracted using a heat engine or steam. The hydrogen on the other hand can be used in a fuel cell to efficiently generate electricity. The byproduct of the aluminum-water reaction is aluminum hydroxide, which is non-toxic and can be easily made back into aluminum. Aluminum hydroxide can also be used for a various number of different engineering applications.

This high density and dual energy output system makes aluminum an incredibly attractive power source. However, the naturally occurring oxide layer on the surface of aluminum protects it from reacting in most environments. Bypassing the aluminum oxide layer safely and efficiently has remained a barrier to putting aluminum-water fuels into practice.

Activation Process for Bulk Aluminum and Reaction with H2O



Figure 2 shows two jars of aluminum BBs undergoing the activation process

Under standard pressures and temperatures, a strong thin clear oxide layer forms on the surface of Aluminum. This layer protects the piece of aluminum from fully oxidizing. However, in Slocum's thesis [1] a novel activation treatment process was discovered that can passivate bulk aluminum using a gallium-indium surface treatment. This process is described in detail in the thesis [1] but is also further analyzed in Peter Godart's thesis.

In brief, Aluminum pieces are activated in a heated liquid gallium-indium eutectic bath and then allowed to sit in a hermetically sealed environment for several days. By applying a gallium indium eutectic, the oxide layer is broken down and seeps into the grain boundaries of the aluminum. When exposed to water, it reacts with the aluminum grains and produces aluminum hydroxide. The sphere is entirely consumed during the reaction, reacting almost to its entirety. This treated aluminum reacts with similar efficiency to activated aluminum powders, but with none of the safety concerns or logistical issues that accompany aluminum powders. This activated aluminum is cost-effective, energydense, safe to handle, and has been shown to work in real engineered systems.

Once the fuel is activated, it can successfully, and reliability react with water by the following chemical reaction.

$$2A1 + 4H2O \rightarrow 3H2 + 2AlO(OH) + Heat$$

This stoichiometric reaction produces both heat and pure hydrogen gas, both sources of energy that can be then converted into a variety of engineering applications. The heat can be used in heat engines, or to generate steam which can be used for steam engines or desalination. The hydrogen gas produced can be used in hydrogen fuel cells or internal combustion engines, to produce mechanical power. As described above, this reaction is incredibly energy-dense, and in many senses is the best way to store hydrogen both weight, volume, and safety-wise when compared against pressure vessels and liquid hydrogen forms.

Aluminum Slurry Form

However, the activated aluminum comes in a bulk form. There is a desire to make the system pumpable. Traditionally, this has been done by powdering the aluminum. Once ground into powder, and before being placed in oil, the aluminum becomes extremely volatile and may begin to react with oxygen or water vapor in the air. For this reason, the powder is ground and stored in argon, however, it is still in contact with the air at various transfer points in the grinding process and remains an insufficient means to work with the fuel.



Figure 3 Diagram of the Silicon Oil separating from the water to surround the aluminum

Instead, it has been shown that an effective way to not only pump the fuel but isolate it from the air is by dissolving it into an oil medium. The ground-up activated aluminum acts as a granular flow being transported in a carrier fluid. This allows for the activated aluminum to be transported with traditional pumping means. When the oil comes in contact with water, it will begin to separate naturally, until no oil remains around the aluminum, and it can fully react.



Figure 4 coating of fumed silica to eliminate clumping

To prevent clumping of the aluminum particles in the silicon oil, Aerosil fumed silica is added to the mixture. This fumed silica when blended thoroughly coats the aluminum particles, and prevents the sticking effect caused by van der Waals forces. To effectively apply the fumed silica, the Aluminum oil mixture must be mixed by a sufficiently high shear rated blender to fully coat the particles and prevent clumping from occurring.

Applications of Technology - Underwater Glider

One application that is a prime application for aluminum fuel technology is underwater autonomous gliders (UAVs). Due to the inherent nature of the vehicle being submerged in a liquid water medium, the craft does not need to carry the extra high-density weight. These water weight requirements limit the feasibility of land or air-based vehicles and are why much of the focus for the Aluminum Fuel system has been around design for compatibility of an underwater platform.



Figure 5 DOUG LE CAD Render from the Spring 2022 MIT 2.014 Cohort

Work is currently underway on a prototype system that one day hopes to integrate the Aluminum Fuel system into a glider. MIT's Spring 2.014 Course has designed and manufactured an underwater vehicle with a substituted lithium battery-based energy system that one day hopes to be replaced by a finished aluminum fuel reactor.

One unique characteristic that is also beneficial to an underwater glider, is the ability to use the hydrogen and heat produced by the reaction for a buoyancy engine. Underwater gliders operate by starting at the surface of the water and then sinking to the desired depth. Hydrofoils on the body of the craft provide lift, and from that lift extend the sinking time and range of the craft. This allows for the craft to naturally extend its range for minimal energy. However, once at depth, the craft must be able to return to the surface to begin another dive cycle. In theory, this can be accomplished through the Aluminum Fuel system. Since the reaction with water produces both hydrogen and heat as a byproduct, this can be used to inflate a bladder on the craft, providing positive buoyancy and returning it to the surface. Once at the surface the craft can

then use the hydrogen to produce electric power since it is now in the presence of oxygen. The craft would then begin another dive cycle with the power generated at the surface.

This novel approach to an underwater glider has the promise to produce a system with unprecedented range and endurance while being green and reusable. This system would have several applications such as monitoring undersea cables, monitoring pollutants in the ocean, and scanning for oil leaks, all while reducing operating costs due to the extended range and endurance. There are numerous other missions and applications for a long-range underwater glider, and the promise of the aluminum fuel system is an incredible match for the design requirements of such a craft.

Aluminum Fuel Production

Process Development using the Colloidal Grinder



Figure 6 picture of the colloidal grinder use to make slurry

To produce a stable slurry, the Aluminum needs to be ground to a finer consistency. Since powdered aluminum is very dangerous due to its high reactivity and energy density, careful consideration needs to be made while producing the slurry.

The slurry process utilizes a colloidal grinder, traditionally used to grind up peanuts in making peanut butter. The aluminum is first activated by applying a surface coat of the gallium-indium eutectic.

This takes the form of many aluminum BBs, small 5mm round spheres. These spheres are then placed in a silicone oil medium and then poured into the top funnel of the grinder. The grinder is bathed in inert Argon gas to ensure no unwanted oxidation from the air and no unwanted reaction to moisture in the air. Since the aluminum is still very reactive in this state, and the grinder produces several grams of slurry, the process needs to be conducted safely and with care. In conducting the process, a four to one ratio by volume of oil to aluminum was used when grinding. This had the dual-use of helping the grinding process, as well as outputting the aluminum already in a slurry form instead of just pellets.

One note is that after a successful production of aluminum slurry, much-activated aluminum is left on the sides and walls of the grinder. This aluminum is still very reactive and must be cleaned up to ensure safety. Firstly, shop towels should be used to wipe down the sidewalls and remove any easy-toreach surfaces. To clean the grinder teeth, it was found that using a pointed file and screwdriver were effective tools to remove the aluminum from in between the gaps. Finally, once all major groupings of aluminum have been removed from the grinder, a small pipette can be used with a bit of deionized water to splash onto the unreacted aluminum. This target is hard to reach aluminum and reacts just a bit to it. This needs to be done carefully, and only after a majority and large chunks have been removed. Finally, a combination of compressed air and shop towels can be used to remove all the aluminum by-products from the machine. Before using the colloidal grinder to produce fuel, it must be checked several times for the absence of activated aluminum to ensure the safety and longevity of the machine.

Due to the long cleanup process, and insufficient yields (much aluminum remains stuck to the walls), other grinders are currently being explored. It might be beneficial for future work to develop an optimized and specific grinder for aluminum fuel, with safety and yield considerations gained from the current device.

Development of a Safety Box



Figure 7 safety box surround the colloidal grinder

One of the first orders of business before any experimentation could take place was to improve the safety of the experimental process. There was an unfortunate mishap during the winter of 2021 that resulted in a lab injury caused by a flash reaction of activated aluminum. This happened when water accidentally came into contact with the colloidal grinder after the production of a bath of Aluminum slurry. This resulted in a substantial injury and a revaluation of the standard operating procedures for fuel production.

Before any research continued, a safety box was designed and manufactured to enclose the colloidal grinder entirely. The box was constructed with 1/8" Polycarbonate, a high-impact-resistant plastic. The polycarbonate provides a good amount of protection to the user if something does begin to spontaneously react inside. The box also had a side port that allowed for a tube fitting to be attached to flood the box with an inert gas to prevent any unwanted reaction to occur. The fuel can react naturally with error due to moisture content in the air.

Originally, the box was going to have three access doors. One on the top of the box for access to the funnel into the grinder, one on the front face of the box to access the power switch, and the last on the

side to remove the container in which the mixed slurry is deposited into. The access door idea was ultimately dropped, due to sealing concerns and complexity. Instead, the holes were covered up by placing a piece of tape over the hole temporarily. Although not an elegant solution, the tape provided adequate sealing to the box, while allowing ease of access inside.

Further design improvements have also been proposed, such as using 8020 framing for the borders and exploring off the shelf sealing doors and solutions. Future work will be done to make the box safer and easier to work with.

High-Pressure Experimentation

Experimental Set-Up



Figure 7 Diagram of the high pressure test apparatus

Since the ultimate goal of experimentation was to test under high pressure, an experimental device had to be engineered to meet specification and safety. Utilizing standard off the shelf tube fittings, the whole system was designed to with stand up to 13MPa.

The system worked as a T, with the aluminum sample placed at the bottom of the T. When the experiment is to begin, the Argon port valve is closed and the water port is open. 10mL of water is then injected into the system using a syringe. The valve is than closed. The pressurized argon valve is then

open, and the pressure is increased until the desired pressure is obtained. A pressure transducer is a fixed at the top of the port to measure the change in pressure. A thermocouple is also attached to the reaction chamber to measure the heat produced by the reaction. Using both the heat and change in pressure, a value for the hydrogen yield is obtained. This is then compared to the maximum stoichiometric yield obtain from measuring the weight of the sample. One note is that it is possible to obtain greater than 100% yield due to the fact that gallium will also react with water.



Figure 8 Swagelok fitting high-pressure testing apparatus

Initial High-Pressure Experiments

Since the goal of this research is to develop a reactor for an Underwater Vehicle, there must be considered into working in a high-pressure environment. The reactivity was measured as a function of initial pressure. The maximum pressure tested was 3.5MPa, which corresponds to a depth of around 350 meters deep. The below graph shows the results of the experimentation.



Figure 9 reactivity of 1 Aluminum BB sphere against initial pressure

The data showed that the reactivity increased as a function of initial pressure. Reactivity was measured as the ratio between hydrogen produced by the reaction, and the stoichiometric maximum that can be produced

Interestingly, the results here showed a different trend than what was previously experimentally tested by Peter Godart during his initial pressure experiments. To explain the increase in pressure a hypothesis was formed. The idea was that in the presence of an initial pressure, naturally occurring bubbles from the hydrogen produces would be suppressed. In theory, the presence of bubbles would introduce gaps in the liquid water, allowing oxygen to permeate into the water. This could potentially passivate the aluminum being reacted and reduce the yield rate.



Figure 10 suppression of bubbles forming which would allow oxygen to come into contact with the aluminum



Argon Flush Experiments

Figure 11 Reactivity versus initial pressure for both air and argon flush experiments

To experimentally confirm this hypothesis, the tests were rerun, but this time by doing a pre-flush with argon. What this looked like was before water was introduced into the test chamber, it was filled with inert Argon gas. Since Argon is much denser than air, the air is flushed out of the system. This removes any

natural oxygen or moisture that might be inside the chamber, ensuring that the only inert gases were present, and the added H2O was the only other reactant.

Results from the experiment show that the hypothesis holds. Firstly, under video analysis of the same experiment in a clear tube, bubble suppression was shown at higher pressures, matching the prediction. Secondly, experimental results showed that there was the same trend for increased reactivity in the presence of higher initial pressure. As assumed, the argon medium average a higher reaction rate overall compared to the air medium, indicating that the hypothesis that the air present did indeed oxidize parts of the sample instead of letting it fully react. Secondly, the argon medium had a much better linear fit correlation compared to the air medium. This makes sense as in the air medium, there is much more random oxidation occurring which can skew the results. When in just the argon medium, the random oxidation from the air is removed.

Conclusions

The Aluminum Fuel reaction at high pressures shows promising results for the adoption of the technology for underwater vehicles. Since the operating environments will be under intense pressure, the system will need to be able to reliably work.

From these initial experiments, a few conclusions can be drawn. The first conclusion is that the fuel is still able to perform under high pressures. This is exciting news as previous experiments showed that the fuel was unable to react at high pressures. The second conclusion is that not only does the reaction still take place at higher pressures, but the reactivity also actually increases. This means the energy yields from the reaction are even better and even more efficient. This is incredibly promising for the feasibility and adoption of aluminum fuel as a viable solution for underwater vehicles.

Future work and testing need to be done at even higher pressures. Due to limitations in the test apparatus and safety concerns when at high pressures, a new test setup will need to be developed. The

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fittings were only rated up to 2000psi, which is still very high, but experiments will hope to be done at even higher pressures. The target pressures for underwater vehicles will exceed 2000psi.

Pumping Experimentations

Geometric Optimization

In Peter Godart and Jason Fischman's paper on Aluminum Slurry design [6], a reactor design was developed but only tested once. This reactor design failed due to the jamming of the fuel while being pumped. To not jam, a few variables can be tuned. The first is inherent to the slurry itself, which is the viscosity of the slurry. Since the slurry is a granular material suspended in a carrier fluid, the viscosity will be determined by the ratio of carrier fluid to aluminum, as well as how homogenous the slurry is. Analytical analysis of the viscosity is coved in detail in their paper. The other two areas of optimization are the pressure provided by the pump and the geometric shape of the pumping. As for the pump, the higher the pressure, the higher the energy need for the pump, so for the sake of scope, a moderately powerful pump was selected for experimentation. The main area of experimentation was chosen to explore how geometric conditions affect the pumping of the slurry. After consultation with Peter and Dr. Ken Kamrin of MIT, the key variable of internal tube diameter, length of mixing tube, and the angle at which the two tubes meet were chosen as key geometric variables to be explored.

Development of Parametric Model for Part Modeling

To rapidly experiment with a few of these geometric hypotheses, a flexible and robust modeling scheme was developed. As mentioned above, when studying the pumping and movement of granular flows, there are a few rules of thumb to follow, but much of the understanding of the behavior of the material can be easily determined experimentally.

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Figure 12 screenshot of the parametric modeling tool developed to quickly iterate through pumping configurations

With iterative experiments in mind, it was determined that the best course of action was to experimentally solve and optimize the best geometry configuration for pumping. To enable these two main processes were leveraged. The first was a novel parametric modeling approach and the second was using additive manufacturing to rapidly-produce said geometries.

The parametric modeling tool produced works by taking a few key geometries, the inner diameter of the pipe, the length of the pipe, and the input angle. The program then solves geometrically and outputs a 3D model that is configured to those parameters. This model not only outputs the base part but also autoupdates every part in the test assembly and outputs the file into a workable file format. This form allowed for very fast and documentable changes in geometry, with minimal headaches to the design process. It also helped ensure similarity between each geometry, ensuring that only the variables desired to be tuned were changed, while the rest of the geometry remained the same.



Figure 13 Build plate of 12 geometries printed at the same time using a high-temperature Formlabs resin

To produce these parts, while keeping the iterative and rapid approach in mind, new cutting-edge 3D printing techniques were used. The two design constraints for producing these parts were the complex and thin inner geometry of the pipes, and the high temperature produced by the reaction. Traditional fixed deposition (FDM) 3D printers print a thermoplastic. This is undesirable for aluminum slurry experimentation due to the poor resolution and surface finish of internal features. FDM printers also print a thermoplastic, which at the temperatures to be seen during the experiment would melt and deform. So instead, a resin SLA-based printer was used to produce the geometries. The resin picked was specifically

chosen for its survivability at high temperatures. The resin printer also allows for very high-resolution internal features needed for the geometries.



Initial Experimentation

Figure 14 Diagram of the various components of the pump experiment

To test the different geometries, the following experimental setup was constructed. Two peristaltic pumps drew fuel and deionized water from two beakers into the printed geometry. The two streams met and then mixed into one output section before being finally pumped into a waste beaker. The two peristaltic pumps were sourced from McMaster Carr, and all the tube fittings and tube diameters were held constant. The water and fuel were pumped together and then met together, mixing and reacting before being deposited into a waste receptacle.

The initial experimentation was designed to be qualitative, a simple go no go for being able to be pumped. The testing was designed to be a starting point before the design of a more rigorous experiment could be done. This is mainly because it was unknown if the Aluminum slurry would even be pumpable. Also, measuring hydrogen yield is a bit further along the line. One needs to develop a working reactor before the yield and efficiencies could be calculated.



Figure 15 pictures of the experimental setup during lab testing

Aluminum/Oil Ratio	Successfully Able to Pump	Notes
65% Al 35% Oil	Jammed	Was not able to flow through the
		entire system
25% Al 75% Oil	Jammed	Was able to pump for a bit before the
		Aluminum began to settle jamming
		the system

Results from the initial experiments proved unfruitful. The Aluminum/Oil Ratio used by Peter Godart and Jason Fischman proved to be insufficient viscosity to pump. The peristaltic pump was able to pull the fuel about 3 inches before the aluminum settled and clogged, not even reaching the peristaltic pump. The two pumps together drew 4.8 W of power and were unable to pump the fuel.



Figure 16 picture of the settled aluminum inside the tube

A workaround to the pumping issues was tried, by increasing the percentage of oil to decrease the viscosity. While this worked, and the fuel was able to pump for a bit, after about 30 seconds of pumping, aluminum began to cling to the sides of the tube and jam it preventing further flow. This prevented the pump from being able to adequately supply more fuel to the reaction chamber.

Conclusions and Future Work

Unfortunately, not many conclusions were reached from the fuel pumping experiments, except that more work needs to be done to validate that a pumpable fuel is even feasible.

Proposed Continuous Flow Reactor Design

Proposed Reactor Design

Looking forward to the future, there is the desire to engineer a working reactor. Initial experiments above did not prove fruitful for successful pumping for various reasons. The first reason is choked points in the tubing, quick settling times, and insufficient pump strength.



Figure 17 Proposed design for a prototype continuous flow reactor

Above is a CAD model of a proposed reactor design, utilizing both the iterative geometry from the experiments and the porous tube reactor model developed by Jason Fischman. The idea is that the tube where the aluminum and liquid water interact is porous, allowing for hydrogen gas to seep through but no water or fuel. Unfortunately, the pumping experiments indicate that this design might not be feasible due to the amount of jamming seen in the pumping experiments, especially with the aluminum tending to clump up on the walls.

A potential solution to this is coating the tubes with material like fumed silica, which will help prevent clumping and friction against the wall. Back pressure on the hydrogen output side will also be needed, to ensure that the hydrogen gas is escaping where it is needed and not recirculating through the system.

One final issue with this system is that the larger pumps will be required to pump the aluminum fuel. The current pumps were already using 5 W of power and were insufficient in pumping the aluminum fuel. Ultimately, this design trade-off will need to be considered when making a reactor as it will ultimately hurt the efficiency.

Oil Recycling Considerations

As noted previously, energy density remains a key design constraint for the system. The introduction of the silicone oil carrier fluid decreases the potential energy density by a lot. The engineering tradeoff of having true pumpable fuel cannot be understated for the mass adoption of this technology. Power engineers and traditional systems are accustomed to working with pumpable and transportable fuels, with a plethora of well-established literature and designs for systems of this nature. To remain a feasible solution, a balance needs to be struck on the ratio of aluminum to the carrier fluid. Presented below is a potential workaround that will be explored in the future.



Figure 18 schematic of potential oil recovery system

Since the silicon oil and water cannot mix and separate in known ways one can take advantage of that for oil recycling. In this proposed design, the waste

However, one downside of this proposed solution is that it is not orientation dependent. The design relies on the fact that the waste will settle on the bottom of the waste tank, with the oil being skimmed off of the top. If this system were to be integrated into an underwater craft as proposed, design considerations would need to be made to ensure that the tank always remained normal to the direction of gravity. This is a potential deal-breaker for this idea, as the extra complexity needed eliminates the orientation independence gained from the pumpable fuel system.

Conclusion

The various experimental and engineering results show promising progress towards the successful engineering of an aluminum slurry continuous flow reactor for underwater vehicles. Firstly, much work was done to update the safety standards and experimental procedures. Since the fuels being worked on are incredibly energy-dense and are also experimental, much work needs to be done upfront to ensure the safety of the scientist doing experiments. Successful updates and safety measures were accomplished for both the production of the aluminum slurry and the clean-up of the tools used to produce a said slurry. The process of developing a homogenous solution was also updated and codified, to help in future experimentations.

Experimentation on reactivity under high pressure proved fruitful, showing a positive correlation between initial pressure and reactivity. Experiments showed that because of the applied pressure, bubble formation is suppressed which helps prevent unwanted oxidation to the pellet to happen. This is very fruitful for the promise of the development of a reactor for underwater vehicles.

Experimentation for a pumpable fuel was less successful. Different combinations of slurries were developed, but all proved to be unpumpable for a sustained period. This was due to either being too viscous to pump or due to the aluminum settling in the piping. Analysis of the pump was also done, showing the power required to operate. If a continuous flow reactor is to be engineered and created the losses in energy density from reducing viscosity and the pumping energy losses must be factored into the design decisions and overall system efficiency. Unfortunately, the best solution might not be using the aluminum in a pumpable form, but maybe some other extruded or batch reactor.

Overall, the promise of using aluminum fuel for underwater vehicles is high, and much of the groundwork has been set for continued experimentation and work to be completed.

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