VULCANIZING UNCURED RUBBER

Vulcanizers, also called heaters, autoclaves, kettles or reactors, are for the curing of rubber and plastic products and composites.

The chemical process that improved the physical properties of natural or synthetic rubber was discovered by Charles Goodyear in 1839. It consists principally of heating rubber with sulfur and other substances (accelerators, carbon black, antioxidants, etc.). The sulfur does not simply dissolve or disperse in the rubber, but rather combines chemically, mostly in the form of cross-links (bridges) between the long-chain molecules; however, the reactions are not fully understood. Vulcanized rubber has higher tensile strength and resistance to swelling and abrasion, and is elastic over a greater range of temperatures. The process of treating crude rubber to improve such qualities as strength and hardness usually involves heating the rubber with sulfur in the presence of moisture in a vacuum in an autoclave.

The autoclave was invented by Charles Chamberland in 1879, although a precursor known as the steam digester was created by Denis Papin in 1679. The name comes from Greek *auto-*, ultimately meaning self, and Latin *clavis* meaning key a self-locking device.

Autoclaves are widely used to cure composites and in the vulcanization of rubber. The high heat and pressure that autoclaves ensure that the highest possible physical properties are repeatably attainable. Aerospace and sparmakers (for sailboats in particular) have autoclaves well over 50 feet long, some over 10 feet wide. Other Applications include rubber rollers, tire re treading, and industrial drum lining.

Principle of Operation

An autoclave applies both heat and pressure to the workload placed inside of it. Typically, there are two classes of autoclave. The process medium can be steam, a combination of steam and air or inert gas. Those pressurized with steam process are used on workloads that can withstand exposure to water, while circulating heated gas provides greater flexibility and control of the heating atmosphere.

Processing by autoclave is far more costly than oven heating and is therefore generally used only when isostatic pressure must be applied to a workload of comparatively complex shape like inflatable rafts. For smaller flat parts, heated presses offer much shorter cycle times. In other applications, the pressure is not required by the process but is integral with the use of steam, since steam temperature is directly related to steam pressure.

The key component of the industrial autoclave is the fast-opening door which is also a critical component in cost of autoclave construction. On one hand, the operator must be able to open and close the door quickly and easily; on the other, the door must satisfy stringent safety requirements.

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Design and Construction

Large steam autoclaves can be exposed to an internal vacuum if the steam fully condenses while the vessel remains sealed. Although external pressure cannot exceed one atmosphere, that can suffice to collapse the vessel and thus significant reinforcing of the autoclave is required.

In unusual situations, the autoclave might have to be square or rectangular instead of round, or it might be vertical instead of horizontal. In an autoclave, as in any machine, all major and minor components should be accessible for inspection, repair, and replacement. Overlook this, and the owner will eventually regret it. {As an example, the autoclave at the Rubber Crafters Plant in Grantsville, WV exploded in 1985 when the door blew off. No one was injured, but for future protection they built a block wall to isolate it.}

Typically, autoclaves use an air duct running across the full circumference of the interior. The duct reduces the diameter of the cylindrical volume by only several inches. To take a typical example, consider an autoclave with an 8-foot internal diameter and a working length of 40 feet. If the interior wall is made of 11gauge (.1196 inch) steel, then it will weigh well over five tons. Heating just the wall itself to an operating temperature of 300 F (149 C) in one hour will take some 90 kilowatts of power. At typical (2000) demand charges, that will cost approximately $2,000 (for a month) in addition to the energy charge (for each cycle). Reducing the wall thickness to 18 gauge will drop this expense by approximately 60 percent. For a saving of $13,000 a year, the average autoclave operator can live with a lot of dents.

On some autoclaves, strange noises come from the inside as they heat up and cool down, caused by distortions in the metal interior as it expands and contracts with the extreme changes in temperature. The interior of the autoclave described above will grow nearly an inch in length during the heat-up part of its cycle. Provision must be made for adequate relief of these movements or they will eventually buckle the interior. If the machine is large, it will require an interior floor adequate to support personnel walking on it, as well as safety devices to protect personnel inside the machine against inadvertent startup.

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Introducing heat into the chamber can be done in a variety of ways. For autoclaves used to process composite parts or perform adhesive bonding, the least costly initially is electric heat. Resistance heaters are compact and can be placed conveniently in the circulating air duct. Since the thermal mass of these heaters is small, control of chamber temperature is precise. These heaters are essentially 100% efficient and can be fitted for any voltage, single or three phase.

Installing more capacity than is required extends the life of the heaters by allowing them to run at lower surface temperatures and it provides greater assurance of attaining required heat-up rates. Increasing the heating capacity generally costs little in initial price. It is unsafe to automatically assume that every autoclave manufacturer uses high quality tubular Incoloy-sheathed rods, individually replaceable and properly supported. In the interest of economy, some expect the customer to accept nichrome wires strung on ceramic insulators.

The drawback to electric heat is operating cost. For a small autoclave operated only periodically, this may not prove to be a major issue. For a mid-size or larger autoclave, the electric bills over the service life of the machine will add up to quite a few times the entire cost of the autoclave.
For example, in the Rochester, New York, where the price of electric energy is four times that of natural gas before taking demand charges into consideration, an autoclave six feet in diameter and twenty-four feet long (with a light-gauge interior wall) would cost about $2,000 a month in demand charges plus an average of $14 an hour in energy when running. The demand charges, incurred as soon as the autoclave is turned on, even if for only a moment, would equal the purchase price of the autoclave in only a few years. Experience with utility bills suggests that this will get worse over time.

The easiest alternative to this is steam heating. This presupposes the presence of a boiler capable of generating steam at high enough pressures to reach the required temperatures. An existing high-pressure steam plant is a fine thing to have and facilitates the use of steam coils, which are simple, compact, and easily controlled. The purchase price of steam coil heating is roughly comparable to that of electric heating, but the operating cost is dramatically lower. If high pressure steam is not available a dedicated boiler for the autoclave is required. The cost can be surprisingly low, making this alternative nearly as economical as direct gas-firing of an internal heat exchanger. It also enables running the autoclave on natural gas, propane, butane, or fuel oil, sometimes interchangeably if the boiler is set up for dual-fuel operation. Where gas supplies are susceptible to interruption, using a small high-pressure steam boiler to run the autoclave and ovens can be a life-saver when dual-fuel firing is incorporated.

Equally economical to operate is an autoclave with a gas-fired heat exchanger built into the pressure vessel. Although this presents some design limitations, it is simpler than using synthetic heat transfer fluids, and of somewhat lower cost. The gas burner assembly is fitted to the far end or the side of the vessel and fires into a heat exchanger inside the air duct. The hot end of the replaceable tube is covered with turbulators for better heat transfer. This recovers the greatest part of the energy of the flue gas. It is simple and reliable, using ordinary natural gas, butane, propane, or other industrial fuel gas.

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The longer the machine, the longer the heat exchanger tube and thus the more efficient it will be. This heating option is less costly than hot oil and more costly than electric or steam (assuming an existing boiler) to purchase, but the extra expense is paid back very quickly. Over its full service life, the electrically heated autoclave will cost enough to have paid for another four or five comparable autoclaves. For any but the smallest lab machines, gas firing and steam heating are, to put it plainly, the best alternatives.

In some circumstances, when steam is available in the plant, considerable money may be saved by using live steam injection. In this approach, the entire interior of the autoclave is filled with live steam at the appropriate pressure. Commonly used in the rubber products industry, this can be adapted to use in curing composites. It requires different vacuum bagging materials but has the advantage of eliminating heaters, ducts, and the circulation fan. With external insulation, there is more room available for workloads, for a given size of pressure vessel. Naturally, this approach presupposes the availability of an appropriately rated boiler.

In certain applications, a low-pressure steam autoclave can replace an ordinary curing oven. The combination of vacuum consolidation, which is equivalent to approximately ten to fourteen psi external pressure, and steam at about the same gauge pressure, will give better results and faster heat-up than the oven would. This approach would be less suitable for materials that have to be brought to curing temperature slowly, since steam transfers its heat fairly quickly compared to even a turbulent circulating air flow. Since the interior of the vessel is repeatedly exposed to steam and then air, over and over, an allowance must be made for corrosion of the vessel walls.

In some circumstances, an externally fired heater brings synthetic thermal fluid to temperatures of 600 F (316 C) to 800 F (427 C), and pumps circulate it through heat exchangers inside the autoclave. This has advantages - gas or oil can be used as a fuel without much concern for the working volume inside the autoclave, and disadvantages - the cost is very high, and it can be trickier to maintain properly. Additionally, it can serve to heat and cool the autoclave by routing the heat transfer fluid through either the heater or the cooling coil, as required by the process.

Cool-down at the end of the process cycle requires a means of extracting heat from the autoclave. The necessity of controlled cool-down depends on the work being processed. With some composite materials in thick lay-ups, slow cooling prevents internal microcracking of the resin matrix resulting from thermally-induced stresses. The cooling method used will depend upon the highest temperature reached before cool-down and the degree of precision that must be maintained as the chamber temperature ramps down.

For low temperatures and cool-down rates that can be allowed to vary significantly or simply cool-down at any rate results from a fixed flow of coolant, water circulated through a coil in the airstream will be effective and inexpensive. In an autoclave operated at high temperatures, special precautions must be taken in cooling. Pumping cold water into a cooling coil at 800 F (427 C) will shorten the coil's life. It also makes it difficult to control the cool-down rate. When cold water hits a heat exchange coil at 800 F (427 C), flash steam is generated, along with mechanical shocks to the system and considerable scaling inside the coil to the extent that the water contains dissolved solids.

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If the highest internal temperature will not exceed 300 F (149 C), then propylene glycol can be used as a heat transfer medium. As propylene glycol is a constituent of food, such as ice cream, there are no toxicity concerns. It has approximately the same specific gravity as water, so pumping is straightforward. Coils don't build up scale deposits. Fluid life is very good if air is kept out of the loop. Propylene glycol must be used without water dilution, and stainless steel plumbing is not necessary. The cost of propylene glycol is not trivial, so the amount of coolant in the loop has to be balanced between the interests of economy and heat dissipation.

It once unpleasantly surprised one large autoclave customer to learn that closed-loop water cooling systems were strictly regulated in his locale. The price tag on this nasty surprise was in the five-figure range. In some localities, dumping pristine and clean cooling water down the drain may be illegal. In general, not using water for cooling can have a number of advantages.

Unless the autoclave uses steam injection, the circulation fan carries the burden of assuring temperature uniformity throughout the working chamber. The purpose of circulating the air or inert gas through the autoclave is to assure effective heat transfer and temperature uniformity which in turn assures process quality and reliability.

Well-designed autoclaves feature removable back ends which provide easy and unrestricted access to the hardware in the unheated area. It is difficult to realize how valuable this is until it is suddenly necessary to remove a sixty horsepower motor that weighs well over half a ton through an opening just barely large enough for it to pass through. Some autoclaves have the circulation fan and motor, mounted in an end-bell of reduced diameter. While this allows the assembly to be removed easily, it also means that the fan is undersized in diameter and thus less efficient.

Below: RCI Smithville Plant - Autoclave on right;
Note tarp covering roof and rebuilt block wall from July 1984 Autoclave explosion