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# Construction of a Mechanically Agitated Autoclave

Stephen A. O'Hare

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# CONSTRUCTION OF A MECHANICALLY AGITATED AUTOCLAVE

by Stephen A. O'Hare

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### A Thesis

Submitted to the Department of Metallurgy in Partical Fulfillment of the Requirements of the Degree of Bachelor of Science in Metallurgical Engineering

> MONTANA SCHOOL OF MINES Butte, Montana June 9, 1955

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#### Introduction

Tungsten is a metal that is very important in our present day industry. It is used as a pure metal, in alloys, and in paints. The greatest contribution of tungsten to our civilization is in the field of ferrous alloys. These alloys also known as high speed steels have the property of maintaining their hardness at a red heat. The ferrous alloys account for approximately 90% of the tungsten consumption in the United States today.

Tungsten is also used in the production of tungsten carbides. Tungsten carbides are very hard and almost impossible to machine by ordinary methods.

Since the beginning of World War II a great deal of investigation has been done on the tungsten deposits in the western United States. These deposits are principally composed of scheelite (CaWO<sub>4</sub>). The scheelite deposits in the western United States are low grade, and desseminated. With the development of nonmetallic flotation methods these vast deposits of scheelite-bearing ores have become a 28901major source of tungsten for the United States, as the high grade ore deposits have become practically depleted.

Since World War II the scheelite ores of the west have been largely treated by leaching with a basic solution

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under elevated temperature and pressure.

Scheelite can be decomposed by sodium carbonate to yield soluble sodium tungstate, and calcium carbonate. When this process is carried out under conditions of elevated temperature and pressure it is known as pressure digestion.

Pressure digestion of scheelite was first carried out commercially in the United States by the United States Vanadium Company, in 1939. The basic patents for the process were issued to Blair Burwell in 1949 and 1950. These patents covered both the process and the pressure vessels.

At the present time pressure digestion appears to be the only way to process the low-grade ores that occur in the western United States.

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### Statement of Problem

Several problems complicated the pressure digestion process. The first of these problems concerns the solubility of sodium tungstate. Sodium tungstate is quite soluble in water, 400 grams per liter, but in pressure digestions the economic limit is approximately 7.5 grams per liter. Beyond this point the reaction is described as slowing down.

The second major problem is the use of a great excess of sodium carbonate. The amount needed is far beyond what is required theoretically. If these two problems could be solved the process would be less expensive and more efficient.

To study these two problems an apparatus was need where a small amount of pure scheelite and sodium carbonate, could be adgitated under conditions of elevated temperature and pressure. The Metallurgy Department of the Montana School of Mines had such an apparatus, but a large amount of scheelite and sodium carbonate would be required to study these problems. I constructed an apparatus so that these problems could be studied.

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### Construction of Pressure Digestion Agitator

In previous work done by Robert R. Beebe on the pressure digestion of scheelite, he used heavy-duty, forged steel 4-in x 3/4-in pipe nipples capped with heavyduty, forged steel caps. These bombs worked very well and there was very few leaks.

Dr. Hames suggested that I try to design an agitator that would use these bombs. This I have attempted to do.

# Details of Construction

Several methods of holding the bombs in place were investigated. The first method tried was to hold the bombs in place by a steel strap placed over the ends. This method held the bombs in place, but they could not be removed individually. I felt that they must be able to be removed separately because tests would be run for different times and probably only one would be removed from the furnace at a time.

Finally I decided that clips of some type would be the only solution to the problem. After investigating several different types of clips one was found that held the bombs tightly but still they could be removed individually.

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The clips were attached to brass plates and the brass plates were attached to a common shaft. Four clips were placed on each brass plate so that two bombs would be on each plate.

Lubricating the shaft proved to be a great problem. At the temperatures that the apparatus would operate ordinary methods of lubrication would fail. Pillow blocks of brass were constructed. I though that the brass would act as the method of lubrication but the shaft showed wear instead of the pillow blocks.

Two pillow blocks of steel lined with babbitt metal were obtained. These two pillow blocks worked well and solved the lubrication problem.

One word of caution should be given concerning the pillow blocks. They should not be used at a temperature higher than 475°F. At temperatures higher than this the babbitt metal will melt.

The pillow blocks are attached to two "A" frames. The "A" frames hold the shaft in place in the furnace.

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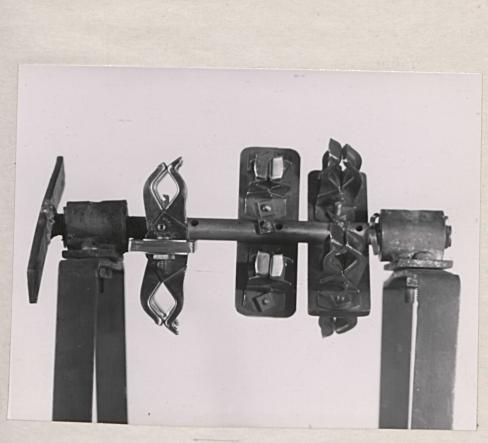


FIGURE A MECHANICAL AGITATED AUTOCLAVE

### Suggestions for Future Work

Future work that may be done with the apparatus for agitation is almost unlimited. While the apparatus was designed for working with scheelite, it may be used for the study of any reactions that occur under conditions of high pressure and temperature.

In the field of pressure digestion of scheelite, the extraction curves that were done by Mr. Robert R. Beebe could be checked and expanded.

There is a possibility that a calcium carbonate layer may form around the scheelite particles. This layer would act as an inhibitor in leaching. Proving that this layer does or does not form is worthy of future investigation.

Finally, the investigation of other basic solutions than sodium carbonate could be investigated.

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