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1. <u>BACKGROUND</u>

Molten aluminum alloys are often described as "universal solvents" they will dissolve most elements, and what is not dissolved is often involved in a chemical reaction that produces complex compounds of oxides, nitrides, carbides and other materials that may or may not be suspended in the melt depending upon their size and density. More than thirty separate contaminant compounds have been found in commercial melts, and these are often times carried into the final manufactured product, with potentially harmful effects.

Probably the most prevalent compounds found are aluminum oxides, which may be present as either films or polygonal particles. The films can range in thickness from 0.1 to 5 microns, and can have diameters between 10 and 1,000 microns. The polygonal particles have been found both separately, and associated with the film oxides. Their size has been reported to vary between 0.2 and 30 microns, with hardnesses high enough to create havoc when encountered by cutting tools in a machining operation.

These oxide inclusions have been the subject of much research, and their formation mechanism and source can be traced by careful examination with sophisticated analytical and microscopic techniques. For example, commercial alloys have been measured to contain between 6 to 16 ppm of oxides of both the alpha and gamma form. The films commonly also contain traces of nitrogen magnesium, iron manganese zinc, sodium, fluorine, potassium and/or calcium. If the alloy contains a significant amount of magnesium, than the dominant oxides will be Al_2Mg0_4 and MgO, and are also present in the form of either films or lumps. When found in castings, all of the above contaminants are almost always associated with hydrogen pores, and are a major source of "porosity" defects, as well as the contained inclusion.

While some of the contaminants may be traced to reduction cells where the aluminum was first reduced from bauxite (e.g. aluminum carbide - A^Q , and AljC[^]), more often than not they originate in a simple reaction with air and the melt, or solution/suspension of surface oxides present on charge materials. When formed in air reactions, the oxides will also be associated with nitrides which may increase the volume and hardness of the films.

2. <u>THE SAMPLING PROBLEM</u>

One of the real difficulties in identifying and quantifying the levels of inclusions present in aluminum melts, is associated with the collection of the initial sample for evaluation. The thirty odd compounds identified in Section 1 have specific gravities ranging from a low of approximately 2.3 to a maximum of almost 4.0. This means that, given enough time, inclusions of sufficient size will either float to the melt surface or settle to the bottom.

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With this range in densities of contaminants, it is obvious that the type of inclusions that will be encountered in castings is somewhat dependent upon the conditions that are present when the casting is poured. If it is made immediately after an aggressive cleaning of the furnace, then some of the more dense inclusions may be mixed into the melt and carried into the castings. If the pouring well or crucible walls are caked with oxide growths, these can almost break off at almost any time and be carried into the mold cavity. If the sludge on the furnace bottom is agitated by the introduction of new metal, then this material can be swept into a position where it can be ladled into the castings. If film oxides are allowed to accumulate in the bath, then these can be found in the final product.

It also stands to reason that if you are sampling to identify the presence of inclusion matter, the extent of sampling required will depend upon the volume of inclusions present, unless the test method collects the contaminants from a large volume of material and displays the accumulate debris. An inclusion problem that results in one percent in the final product, is a lot more difficult to detect than a problem contributing fifteen percent scrap, and will require more sampling to identify than the larger problems.

3. <u>MEASUREMENT METHODS</u>

Over the years, some very sophisticated measurement techniques have been developed to assess the level of contamination. These range from complex electronic devices that characterize the volume, size and distribution of paniculate materials suspended in the melt (e.g. the 4-M method or LIMCA devices), to the Straub-Pfieffer vacuum solidification test. The latter test is at present the industry standard for measuring hydrogen levels in foundry melts, but also produces information on the level and volume of inclusions present. This is gained by observation of solidification of the sample — when inclusions and oxides are present, bubbles will develop in the melt at a very early stage, and may be seen coming out of the melt as it solidifies.

Other methods commonly used by researchers include filtration of the melts, (e.g. the PODRA and UCAR LAIS tests) and the collection of the contaminants for subsequent examination and characterization. Some of the tests have been made dynamically quantitative by coupling load cells to the devices to sense either the rate of accumulations of debris on the filter, or the transport rate of material filtered. Thus, a short term indication is available of the melt quality, and corrective action may be taken to improve melt cleanliness.

The problem of actual quantification of the volume of contaminants is complex, and has also been approached by some researchers by methods which include complete solution of the sample material, with collection of the insolubles. The solution rate has been accelerated by the use of mixed acids or solvents, and even the imposition of an electric current. Unfortunately, many of the compounds and contaminants present are also partially soluble in the solutions used, and are thus lost from the sample. Another difficulty is the physical problems of dissolving the sample itself — which usually limits the sample size to a few grams.

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4. FRACTURE TESTS

Fracture tests have long been recognized to offer many of the features considered to be desirable for a shop floor test:

- **A.** Low Cost: The tooling and equipment required to pour a test coupon is minimal, and does not require a significant investment in either first cost or continuing evaluations.
- **B.** Quick Results: The results are readily available on the shop floor, and do not require lengthy testing or evaluation in remote laboratories.
- **C.** Low Level of Operator Skilled Required: The techniques of pouring the test shapes can be quickly taught to anyone experienced in working with molten metal. Reproducible results can thus be generated, within the limitation of the volume of material being sampled.
- **D.** Sample Retention: If problems are identified on the fracture tests, the samples may be retained for more careful evaluation and analysis with more sophisticated techniques. Although, the mere identification of a problem should be enough to prompt the initiation of corrective action (fluxing, degassing, or filtration) to remove the problem materials.

That is not to say that fracture testing does not have some serious drawbacks, which have limited its application up to now. The drawbacks include:

- **A. Inclusions Location:** In order for a fracture test to identify the presence of inclusions, it is necessary that the fracture plane traverse the inclusion. The probability of this occurring is proportional to the number of fracture planes, and the volume fraction of inclusion matter that is present.
- **B.** Non-Quantitative Nature of Tests: The tests do not lend themselves to quantification, and are thus difficult to use on a go no go basis as a quality control test.
- **C.** Interpretation of Results: While the presence of foreign matter can be seen easily on a fracture face, the interpretation and identification can require experience to insure that the proper corrective action is taken. For example, shrinkage or high levels of gas can cloud the fractures and sometimes can be mis-identified as inclusions.

Many of the fracture coupons used to date have consisted of round shapes that are either bent until fracture, or are notched and then broken on the induced plane. All suffer from the limitations listed above to varying degrees, and have not found wide acceptance in the aluminum foundry industry.

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5. <u>K-MOLD TEST</u>

The K-Mold test is a fracture test that was developed by researchers at Nippon Light Metals, and is presently specified, both as a melt qualification test on incoming raw materials, and as a process control tool in casting operations of Japanese owned foundries.

The shape of the test piece is a flat plate, as opposed to a found test coupon, and is interrupted by four knife edges on the cope side of the plate. These edges serve both as fracture plane surfaces, and also as collectors to concentrate inclusion matter present in the molten sample being evaluated.

While the geometry of the shape is quite simple, there are many subtleties in the design which influence the efficiency of the mold to produce clear and readable indications.

In use, multiple samples of the plate are poured from the metal being evaluated, and are fractured immediately by the technician performing the test. Those fracture surfaces containing inclusions are considered to be an "event", and are expressed as a ratio of events to sound surfaces. For example, if two bad fractures are found in a total of twenty fractures, the ratio is 2/20 = 0.1.

Allowable ratios are established for each product family, with those demanding a higher level of metal purity specifying a lower level of "K-Value". Typically, a thin walled casting that has pressure tightness requirements may require maximum values of 0.05 (a maximum of one inclusion in twenty fractures). Heavier walled castings with less critical requirements may be produced to higher K-Values.

While certainly not a perfect test the use of the K-Mold does seem to answer many of the needs of the industry for a method of melt quality evaluation. The results produced are at least semi-quantitative, are quickly available for corrective action to be taken on the shop floor, and may be performed by a person with minimum level of training and experience. The drawbacks lie principally in the limited volume of material actually being sampled, and the possible sensitivity of the test to operator technique.

The test is presently being used by several researchers and commercial foundries in this country, and is being evaluated to identify possible defects areas for further refinement of the method. A library of defect indications is also being assembled to simplify and assist operator training.