**Fig. 1a**

- **Composition**: Modified 7075-type for temperatures and pressures stated below.
- **Temperature**: 1250 °F, minimum to approximately 1350 °F maximum.
- **Crucible Composition**: Non-reactive, alloy impurity-free, moisture/hydrocarbon-free. (e.g. High purity graphite)
- **Alloy Temperature**: 1250 °F, minimum.
- **Mold Temperature**: 1200 °F, minimum.
- **Mold Composition**: Non-reactive, alloy impurity-free, moisture/hydrocarbon-free. (e.g. Machined high purity graphite, molded carbonaceous-bonded refractory oxide powder particles, etc.).
- **Initial Alloy Temperature**: 1200° F, minimum.
- **Cooling Rate**: Controlled so that alloy solidification front (alloy at temperatures in the range of 1175° F to 900° F) is moved progressively, incrementally and directionally from alloy/mold points farthest from casting riser to casting riser.
- **Alloy Stopping Temperature**: 800° F, maximum to 600° F minimum.

**Atmosphere**: Cyclically controlled: (l) from initial ambient to 5 x 10^-3 mm Hg to above standard atmospheric pressure (e.g. 15 psig.) with dry inert gas (e.g. argon) at least twice prior to cooling any metal below 1200° F and (2) thereafter to 5 x 10^-3 mm Hg to above standard atmospheric pressure (e.g. 15 psig.) with dry inert gas (e.g. argon) at least once while metal is in temperature range of 1025° F to 1150° F.

**Pressure**: Controlled as indicated immediately above.

**Atmosphere**: Controlled - non-reactive, hydrogen-free, moisture/hydrocarbon-free (e.g. argon, <100 p.p.m. H2O or hydrocarbons)

**Temperature**: 600° F, minimum to 800° F maximum.

**Method of Removal**: Ejection of casting from mold.

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**REMOVE CASTING FROM MOLD**

- **Atmosphere**: Ambient
- **Pressure**: Ambient
- **Temperature**: 600° F, minimum to 800° F maximum
- **Method of Removal**: Mold disintegration by oxidation.

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**ANNEAL**

- **Atmosphere**: Ambient
- **Pressure**: Ambient
- **Temperature**: 500° F, minimum to 600° F, maximum
- **Time**: Sufficient to develop ductility at temperature. (e.g. 5 hours at 500° F)
Atmosphere: Ambient.
Pressure: Ambient.
Starting Temperature: 600°F, minimum, to 800°F, maximum.
Cooling Rate: Conventional.
Final Temperature: Ambient.

**2nd STAGE COOLING**

Atmosphere: Ambient.
Pressure: Ambient.
Starting Temperature: 500°F, minimum, to 600°F, maximum.
Cooling Rate: Conventional.
Final Temperature: Ambient.

**REMOVE CASTING FROM MOLD**

Atmosphere: Ambient.
Pressure: Ambient.
Temperature: Ambient.
Method of Removal: Ejection of casting from mold or disintegration of mold.

**SOLUTION AND HOMOGENIZATION HEAT TREATING**

Temperature: 860°F ± 40°F
Time: Sufficient at temperature to eliminate second phase precipitates at grain boundaries (e.g. 24 hours at 860°F per 0.1" metal thickness).

**POST-HEAT TREAT COOLING**

Initial Temperature: Heat treating temperature.
Cooling Rate: Sufficient to obtain non-equilibrium metallurgical structure (e.g. Quench in ambient temperature water).
Final Temperature: Ambient.

**AGING**

Temperature: Sufficient with time to harden alloy to optimum extent (e.g. 450°F to 250°F from corresponding 2 hours to 24 hours).
Time: Sufficient with temperature to harden alloy to optimum extent (e.g. 2 hours to 24 hours at corresponding 450°F to 250°F).
Initial Temperature: Aging temperature.
Cooling Rate: Conventional.
Final Temperature: Ambient.
ABSTRACT OF THE DISCLOSURE

A method for solidifying molten aluminum alloys wherein the molten material is subjected to environmental cycling in a closed chamber both prior to and during molten metal solidification. The processing prior to molten metal solidification involves cycling the environment of a closed chamber containing the metal and mold before cooling any of the molten metal below the alloy liquidus temperature (e.g., prior to cooling below 1175° F. in the case of modified 7075-type aluminum alloy): (1) first to a vacuum pressure condition such as approximately 3 × 10⁻⁴ mm Hg absolute, and (2) afterwards to an above standard atmospheric pressure condition using a dry, non-reactive gas (e.g., to 15 ps.i.g. with pure argon or nitrogen). Also, the processing prior to molten metal solidification preferably involves repeating the stated sequence of steps at lease once. The subsequent processing during molten metal solidification involves repeating the stated sequence of steps, preferably at least once, after the metal has been partially solidified by cooling to a temperature in the temperature range that extends from the alloy liquidus temperature to the alloy solidus temperature. In the case of modified 7075-type aluminum alloys, by way of example, the repeated steps are accomplished at a metal temperature in the range of 1025° F. to 1150° F. Solidification is then completed in the repressurized, non-reactive environment by cooling the alloy to a temperature below the alloy solidus temperature and afterwards the casting is ejected from the mold and heat-treated to develop optimum properties. In the heat-treating it is preferred that the solidified alloy be subjected to a vacuum condition such as 2 × 10⁻⁴ mm Hg at a temperature in the range extending from the alloy solidus temperature to the alloy solid solubility limit temperature a sufficient time such as 24 hours per 0.1" of casting thickness to obtain proper solutioning and homogenization. Afterwards, the casting is preferably immediately quenched and subjected to post heat-treat aging, such as on an accelerated basis by heating at from 250° F. to 450° F. for from 24 hours to 2 hours for modified 7075-type aluminum alloys.

DESCRIPTION OF THE DRAWING

FIGS. 1a and 1b comprise a sequential flow diagram for a controlled directional solidification and heat-treating process using the method steps of this invention.

TABLE I

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Modified 7075-Type</th>
<th>Alloy 220</th>
<th>Experimental Alloy W</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zinc</td>
<td>6.19</td>
<td>&lt;0.10</td>
<td>4.5</td>
</tr>
<tr>
<td>Copper</td>
<td>1.18</td>
<td>&lt;0.10</td>
<td>1.5</td>
</tr>
<tr>
<td>Titanium</td>
<td>0.15</td>
<td></td>
<td>0.17</td>
</tr>
<tr>
<td>Beryllium</td>
<td>0.11</td>
<td></td>
<td>0.11</td>
</tr>
<tr>
<td>Chromium</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Magnesium</td>
<td>0.01</td>
<td></td>
<td>0.01</td>
</tr>
<tr>
<td>Iron</td>
<td>0.00</td>
<td></td>
<td>0.08</td>
</tr>
<tr>
<td>Aluminum</td>
<td>0.09</td>
<td>0.04</td>
<td>0.08</td>
</tr>
</tbody>
</table>

Total Liquidus temp (°F) 1275.0 1275.0 1275.0
Solids temp (°F) 975 975 975

Such compositions are stated on a percentage by weight basis.

The preferred procedure for processing the aluminum casting alloy composition of Table I to obtain increased tensile strength properties in the as-cast, heat-treated, and aged condition and in accordance with this invention involves the steps of FIGS. 1a and 1b numerically designated 111 through 123. The melting and pouring steps referenced as 111 and 112 and the aging and post-aging cooling steps referenced as 122 and 123 in the process are entirely conventional. However, important differences over conventional practices have been developed with respect to steps 113 through 121. It should be noted that alternate sequences are available with respect to the processing intermediate blocks 113 (First Stage Cooling) and 120 (Solution and Homogenization Heat-Treating). Different of the alternate sequences may be preferred depending on hereinafter-described limitations; in any event, the desired end-results are obtained regardless of which one of the disclosed alternate sequences is selected.

The melting step referenced as 111 may be accomplished in a normal ambient air environment. The aluminum casting alloy to be cast should be heated to appreciably above the alloy liquidus temperature; in the case of the 7075-type alloy of Table I, a temperature in the range of 1225° F. to 1350° F. attained prior to pouring is generally satisfactory. It is highly desirable that the crucible apparatus utilized for melting be non-reactive with respect to the alloy and also be free of included or occluded alloy impurities, water, and hydrocarbons. De-gassed, high-purity ATI graphite is a satisfactory crucible material.

Pouring step 112 may also be accomplished within a normal ambient oxygen-containing air environment. In accomplishing pouring, the alloy temperature is generally not less than the minimum temperature of melting step 111. The casting mold into which the molten alloy is poured should be, as in the case of the crucible apparatus referenced above, non-reactive with respect to the alloy and should be free of impurities, moisture, and hydrocarbons, whether included or occluded. The above-suggested high-purity graphite, provided with a machined internal cavity and riser configuration, is a suitable mold material. Also, refractory oxide particles may be molded to the desired configuration and afterwards carbonaceous bonded to form a non-reactive casting mold. As indicated by co-pending application Ser. No. 703,206, filed Jan. 11,
1968, -325 mesh zircon flour mixed with a conventional thermosetting phenolic resin may be molded with the required internal cavity and riser configuration and afterwards properly fired to develop a satisfactory casting mold. It is preferred that the casting mold be heated to a temperature near or above the alloy liquidus temperature (in the case of the Table I 7075-type alloy, to a mold temperature of at least approximately 1200° F.) at the time of pouring, particularly if thin-walled casting configurations are involved. Mold heating may be accomplished through the use of cooperating electrical resistance heating elements secured to the casting mold in heat-transferring relation.

First-stage cooling, the step referenced by block 113, is necessarily accomplished in a sealed chamber and involves a time interval from after completely filling the mold casting and riser cavity with molten metal to completing metal solidification by cooling to a temperature below the alloy solidus temperature. The first-stage cooling accomplished in step 113 preferably occurs within a controlled chamber interior providing a metal/mold environment that is adhered to prescribed conditions of pressure and composition.

After the molten metal and casting mold are placed in the chamber and the environment sealed, the interior pressure is reduced from ambient conditions to a vacuum condition such as approximately 5 x 10^-4 mm. Hg using conventional vacuum pump, booster blower, and cold trap equipment. Next, the chamber interior is repressurized to a pressure in excess of standard atmospheric pressure (e.g., to 15 psig) employing a non-reactive gas medium such as dry, pure argon or nitrogen with a hydrocarbon, hydrazine, and moisture content preferably to six parts per million or less. It is preferred that such evacuation pressureization steps be repeated through at least one additional cycle before the molten metal (and casting mold) is reduced in temperature to below the alloy liquidus temperature. In the case of a Table I alloy, the necessary cycles are completed before any metal has been cooled to below approximately 1200° F.

Step 113 also involves further environment cycling after the molten metal has been partially solidified, as by directional solidification, to a temperature in the range extending from the alloy liquidus temperature to the alloy solidus temperature. In the case of modified 7075-type aluminum alloy, the sequence of steps given above is accomplished preferably at least once when the metal has been solidified to a temperature in the range from 1025° F. to 1100° F. to cast alloy system environment cycling steps are accomplished at a temperature in the upper portion of the specified liquidus to solidus temperature range.

It is particularly important in castings having a configuration with a Shape Factor substantially in excess of 1.0 that the cooling accomplished during step 113 be accomplished so that the alloy solidification front moves progressively from the casting portion furthest from the included riser to the riser. Co-pending application Ser. No. 652,392 assigned to the assignee of this application discloses an apparatus that may be operated to obtain the desired rates and directionality of cooling. Step 113 involves continued cooling in the repressurized environment to a temperature below the alloy solidus temperature prior to ejection of the casting from the mold. Normally such continued cooling to complete solidification continues until the molten metal is cooled to approximately 800° F. but not below approximately 600° F. for purposes that are hereinafter described.

The process steps utilized immediately subsequent to composite step 113 but prior to solution and homogenization for the Table I aluminum casting alloys, the latter being heat-treated and aged after removal from the mold. Heat-treating is preferably accomplished essentially in accordance with the type of environment specified for step 120 wherein a vacuum of $2 \times 10^{-4}$ mm. of mercury (absolute) is utilized. The preferred tempera-
ture for heat-treating step 120 will vary in relation to the alloy composition. In the case of the modified 7075-type material, 860° F.±40° F. is generally preferred. Alloy 220 can be heat-treated at a temperature in the range of 800° F.±40° F.; 950° F.±40° F. is considered satisfactory for heat-treating the other alloy detailed as to composition in Table I. For the alloys specified in Table I, 24 hours at temperature per 0.1" of metal thickness is generally satisfactory from the standpoint of time. Step 120 is accomplished so as to minimize the presence of entrapped hydrogen and second-phase precipitates at alloy grain boundaries.

The post-heat-treat cooling step 121 preferably occurs at cooling rates that are sufficiently high to obtain a nonequilibrium metallurgical structure. It is also preferred that the alloys of Table I be immediately quenched from the solution and homogenization heat-treating temperature to ambient temperature in ambient temperature water, equipment permitting. This differs from the normal practice of accomplishing post-heat-treat cooling (step 121) in heated water such as 150° F., for instance, and with an intermediate heating following cooling in air.

The steps identified by blocks 122 and 123 are conventional accelerated aging steps. In terms of the aluminum alloys of Table I, accelerated aging temperature-time histories in the range of from 450° F. for 2 hours to 250° F. for 24 hours are adequate. No environmental controlled atmosphere or environmental controlled pressure is required. Cooling in accordance with step 123 is conventional and may be accomplished by immersing the acceleration-aged alloy in the ambient temperature environmental atmosphere.

A casting alloy having a composition corresponding to the modified 7075-type alloy set forth in Table I was processed in accordance with the steps set forth in Figs. 1a and 1b of the drawings, using the alternate sequence of blocks 114 through 116. The molten cavity incorporated the external configuration of a missile fin and included a joining riser region positioned above the fin configuration region. The completed casting was heat-treated as specified in block 120 using a temperature of 860° F. for 48 hours. Since the available heat-treating equipment did not permit quenching directly from step 120, the part was air cooled after homogenization heat-treating and afterwards heated to 860° F. in an air furnace for 2 hours and then water-quenched. Round tensile test bars were machined from coupons cut from the casting and were found to have the properties set forth in the following Table II:

<table>
<thead>
<tr>
<th>Table II</th>
<th>Ultimate tensile strength</th>
<th>Yield strength</th>
<th>Percent elongation</th>
<th>RB hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Top</td>
<td>71,390</td>
<td>66,100</td>
<td>2.6</td>
<td>82.5</td>
</tr>
<tr>
<td>Do</td>
<td>72,600</td>
<td>66,200</td>
<td>2.8</td>
<td>85.3</td>
</tr>
<tr>
<td>Middle</td>
<td>72,790</td>
<td>65,300</td>
<td>2.8</td>
<td>85</td>
</tr>
<tr>
<td>Do</td>
<td>72,950</td>
<td>65,500</td>
<td>2.7</td>
<td>85</td>
</tr>
<tr>
<td>Bottom</td>
<td>73,300</td>
<td>66,600</td>
<td>2.5</td>
<td>85.3</td>
</tr>
<tr>
<td>Do</td>
<td>74,800</td>
<td>66,900</td>
<td>2.1</td>
<td>85.4</td>
</tr>
<tr>
<td>Do</td>
<td>75,100</td>
<td>66,200</td>
<td>2.4</td>
<td>84</td>
</tr>
</tbody>
</table>

The completed and heat-treated casting was examined radiographically and found to be free of observable defects, particularly as to pin-hole porosity. Included hydrogen was minimal. The improved physical and metallurgical properties are attributed, at least in part, to the invention claimed herein. In this regard it should be noted that pressures considerably in excess of standard atmospheric pressure are considered desirable for repressurization purposes. Although the specific example involves a repressurization to 15 psig, pressures substantially above the level are considered to have likely beneficial effects. Vacuum pressure levels are selected with reference to the apparent volatility of alloy constituents at the metal temperature involved and so as to preclude alloying constituent depletion due to processing requirements.

Claim:
1. A manufacturing method comprising the steps of:
   (a) Confining an aluminum alloy at a temperature above the alloy liquidus temperature in a sealed chamber having an interior environment,
   (b) Cycling said chamber environment to a vacuum pressure condition and then using a non-reactive gas to a pressure condition above standard atmospheric pressure while said aluminum alloy is at a temperature above the alloy liquidus temperature,
   (c) Cooling said aluminum alloy to a temperature in the range from the alloy liquidus temperature to the alloy solidus temperature to partially solidify said molten aluminum alloy,
   (d) Cycling said chamber environment to a vacuum pressure condition and then using a non-reactive gas to a pressure condition above standard atmospheric pressure while said partially solidified aluminum alloy is at a temperature in the range from the alloy liquidus temperature to the alloy solidus temperature,
   (e) Cooling said aluminum alloy to a temperature below the alloy solidus temperature to completely solidify said aluminum alloy prior to removal from said sealed chamber.

2. The method defined by claim 1, wherein said cycling of said chamber environment is to a vacuum pressure condition of approximately 5×10⁻³ mm. Hg. and to a pressure condition above standard atmospheric pressure of at least approximately 15 psig.

3. The invention defined by claim 1, wherein said cycling of said chamber environment while said aluminum is at a temperature above the alloy liquidus temperature involves at least two such cycles.

4. The invention defined by claim 3, wherein said cycling of said chamber environment while said partially solidified aluminum alloy is at a temperature in the range from the alloy liquidus temperature to the alloy solidus temperature involves at least two cycles.

5. The invention defined by claim 1, wherein said cycling of said chamber environment while said partially solidified aluminum alloy is at a temperature in the range from the alloy liquidus temperature to the alloy solidus temperature is accomplished at a temperature in the approximately upper half of said range.

6. The invention defined by claim 1, wherein said method includes a subsequent step of solution and homogenization heat-treating said completely solidified aluminum alloy, said subsequent step being accomplished in an environment having a vacuum pressure condition and at a temperature in the range from the alloy solidus temperature to the alloy solid solubility limit temperature.

7. The invention defined by claim 6, wherein said environment has a pressure condition of approximately 2×10⁻⁴ mm. Hg.

8. In a method of solidifying a molten aluminum alloy, the step of subjecting a surface of said alloy alternately (a) to a vacuum pressure condition and (b) to a hydrogen, hydrocarbon, and moisture-free non-reactive gaseous atmosphere condition substantially above standard atmospheric pressure prior to cooling said alloy to below the alloy solidus temperature.

References Cited
UNITED STATES PATENTS
3,369,591 2/1968 Welch et al. 75—141 X
L. DEWAYNE RUTLEDGE, Primary Examiner
E. L. WEISE, Assistant Examiner
U.S. Cl. X.R.
75—135, 141, 142, 146, 147; 148—3, 159; 164—122