Dec. 30, 1969 M. J. PRYOR ET AL 3,486,947

ENHANCED STRUCTURAL UNIFORMITY OF ALUMINUM BASED ALLOYS BY THERMAL TREATMENTS

Filed June 21, 1967 2 Sheets-Sheet 1

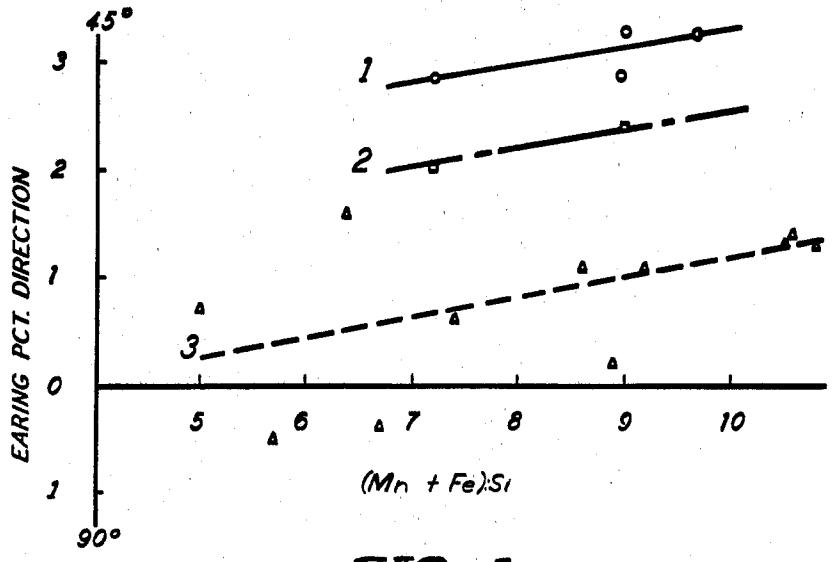


FIG-1

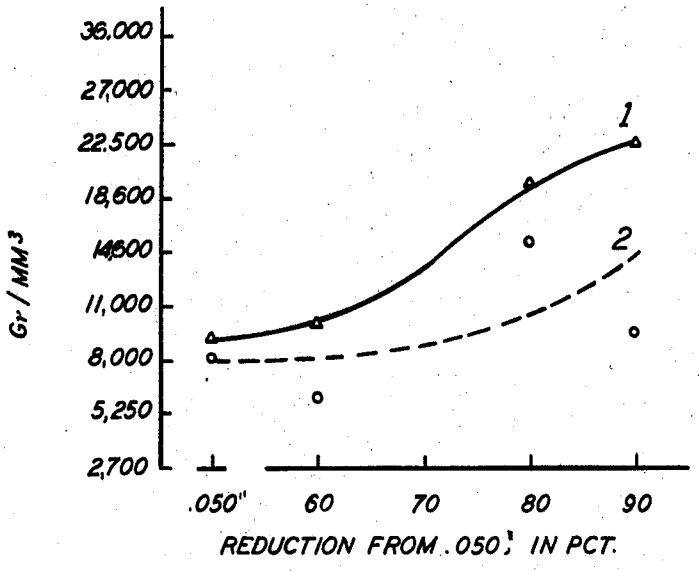


FIG-4

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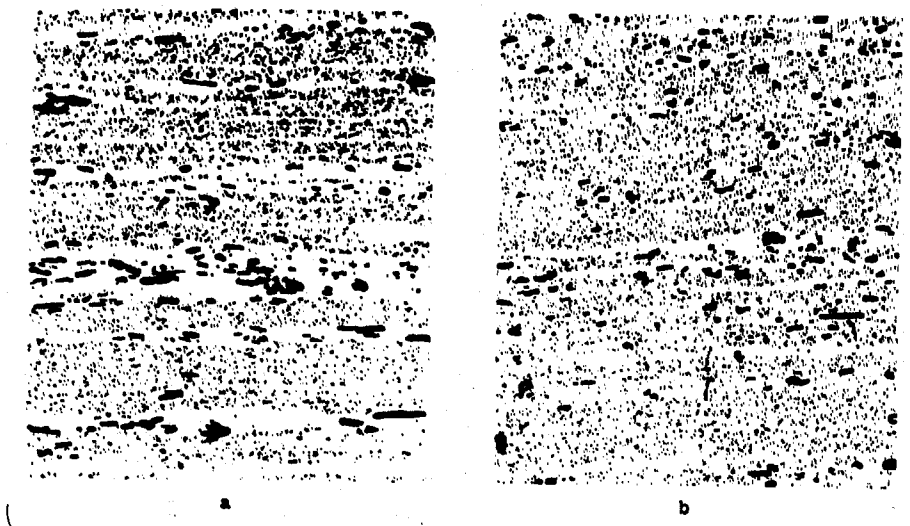


FIG - 2

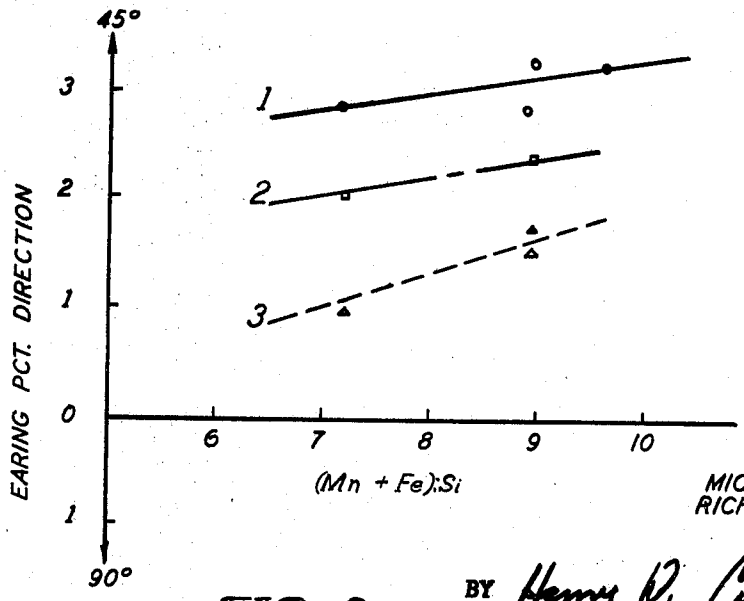


FIG - 3

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ENHANCED STRUCTURAL UNIFORMITY OF ALUMINUM BASED ALLOYS BY THERMAL TREATMENTS

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U.S. Cl. 148—11.5 8 Claims

ABSTRACT OF THE DISCLOSURE

This invention is directed to a method of treating aluminum base alloy castings containing from about 0.5 to 2.0% manganese in order to obtain fine grain size and very low earing characteristics comprising: heating the castings at a temperature of 950 to 1150° F. for 5 to 25 hours, cooling at a rate of between 50 and 200° F. per hour to a temperature of from 700 to 950° F., hot working from 10 to 70% within a temperature range of from 700 to 950° F., reheating the worked article to 950 to 1100° F. and holding within this temperature range for 5 to 25 hours, cooling at a rate from 50 to 300° F. per hour to 700 to 950° F., hot rolling at least 75% at a temperature in the 700 to 950° F. temperature range, cooling to room temperature, annealing at a temperature from 550 to 800° F. for a period of time of from one minute to 24 hours, cooling to room temperature, cold rolling at least 10%, and annealing to recrystallize the alloy.

In the processing of aluminum alloys containing from 0.5–2.0% manganese, the molten metal is directly cast into ingots. In ingots of commercial size, a microstructure as follows is obtained. The primary aluminum precipitates out of the liquid first until a eutectic valley is reached, where precipitation of the eutectic aluminum and (Mn, Fe)Al_6 occurs. Eutectic precipitation continues until a peritectic point is reached where, according to the equilibrium diagram, a peritectic reaction should take place.

However, under the non-equilibrium conditions existing in commercial casting, the peritectic reaction is almost totally suppressed. Furthermore, the eutectic which forms after the primary aluminum has precipitated exhibits divorcement, that is to say, the aluminum portion of the eutectic precipitates on the previously precipitated primary aluminum, leaving the (Mn, Fe)Al_6 to solidify separately. Because of the cooling rate imposed by ingot size, the structure consists of a series of large dendritic cells, often 25 microns and greater in diameter containing the primary aluminum as a core, outlined by the (Mn, Fe)Al_6–Al eutectic.

It is conventional to preheat and hold such manganese containing aluminum alloys at elevated temperatures prior to hot rolling in order to obtain a more uniform structure.

This treatment, generally known as the preheat treatment, is carried out between 950–1150° F. to equalize short range compositional gradients, promote completion of unfinished solid state reactions and obtain a more uniform structure within the ingot, by the process of diffusion. Upon cooling the ingot from the preheating temperature, precipitation of the manganese constituents take place which, ideally would be uniform precipitation of the manganese constituents is not obtained in this step, a banded structure will develop during subsequent hot working. This banded structure cannot be eliminated by cold working and annealing, and will result in coarse and variable grain size, together with high earing percentages in subsequent forming operations. Thus, the structural conditions during preheating which lead to the development of this banded structure must be avoided.

Since precipitation of manganese constituents involves a sluggish diffusion reaction the times required for banding elimination by preheat treatment alone are abnormally long. Furthermore, with very thick cast ingots and, therefore, very coarse dendrite spacings, diffusion distance becomes unusually long. Effective thermal treatment in the absence of additional thermo-mechanical steps for obtaining structural uniformity at these temperatures, therefore, requires unusually long holding time for 20" thick commercial ingot of from 50–100 hours. Such long holding times are not economical commercially.

Several attempts have been made previously to shorten the high temperature holding period by subsequently employing various auxiliary thermal and thermal-mechanical treatments. Typical process sequences are described in U.S. Patents Nos. 3,249,491, 3,219,492 and 3,304,208. All of the processing described in this art involves shorter high temperature holding times which are invariably succeeded by very slow cooling at rates less than 50° F./hour to the hot rolling working temperature which is normally within the range of 700–950° F. for these alloys.

Even with these very slow cooling rates extremely fine grain size, smaller than 8,000 grains/mm.^{3} is not always assured. In general, the application of these ultra slow cooling rates is not economical commercially because of the extended cooling times required. Furthermore, these references are not concerned with the production of a product having low earing characteristics as is the present invention.

It is an object of this invention to process ingots containing a large dendritic cell size to wrought manganese containing alloy products having fine grain size and low earing characteristics.

It is another object of this invention to treat thick chill-cast aluminum alloy ingots containing Mn having a coarse divorced eutectic microstructure in order to obtain wrought products which exhibit fine grain size and low earing characteristics.

It is another object of this invention to process aluminum base alloys containing manganese to deep drawing products having a fine grain size and low earing characteristics with greatly foreshortened high temperature holding times so that the processing is commercially feasible.

It is another object of this invention to process aluminum base alloys containing about 0.5 to 2.0% manganese to deep drawing products having a fine grain size and low earing characteristics with greatly shortened high temperature holding times and relatively rapid cooling rates to the hot working temperature so that the processing is commercially feasible.

Other objects will appear from the following description and claims.

The following process, which is economical commercially, has been found to achieve the additional objects

of avoidance of banding, fine grain size and low earing characteristics. The process is as follows:

(1) Heat the alloy at a temperature of 950 to 1150° F. for 5 to 25 hours;

(2) Cool at a rate of between 50 and 200° F. per hour to a temperature of from 700 to 950° F.;

(3) Hot work from 10 to 70% within a temperature range of from 700 to 950° F.;

(4) Reheat to 950 to 1100° F. and hold at this temperature for 5 to 25 hours;

(5) Cool at a rate from 50 to 300° F. per hour to 700 to 950° F.;

(6) Hot roll at least 75% at a temperature in the 700 to 950° F. temperature range;

(7) Cool to room temperature;

(8) Anneal at a temperature from 550 to 800° F. for a period of time from one minute to 24 hours;

(9) Cool to room temperature;

(10) Cold roll at least 10%;

(11) Anneal at 550–800° F. one minute to 24 hours to ensure recrystallization;

(12) Additional cold rolling and annealing treatments may be provided subsequently as desired in order to give the desired gage.

It can be seen from FIGURE 1 that dendritic cell size generated in casting has a strong influence on earing. Curves **1** and **2** are for a commercial size ingot in which the dendritic cell size was from 25 to 40 microns. Curve **3**, however, is for a laboratory cast ingot with a considerably smaller dendritic cell size of less than 10 microns. It is apparent that if the dendritic cell size is small, as is the case in curve **3**, earing is considerably less than in curves **1** and **2** where the dendritic cell size is larger. It is apparent from FIGURE 1 that if low earing (below 1%) is to be obtained, the large commercial cell size must be efficiently broken up.

Furthermore, the curve demonstrates the tendency of reduced earing with lower values of the (Mn+Fe):Si ratio.

FIGURE 2 is in the form of two photomicrographs of alloy 3003 containing 1.06% mn, 0.57% Fe and 0.23% Si. FIGURE 2A the banded structure which results from a single hot rolling step at 850° F. following a preheat treatment of 1150° F./5 hrs. and 50° F./hr. cooling. This is to be contrasted with FIGURE 2B wherein after identical preheat treatment, hot rolling was performed at 850° F. for only a 30% reduction, followed by a reheat treatment to 1100° F. for six hours and subsequent cooling to 850° F. at a fast cooling rate of 100° F. per hour followed by a second hot rolling step at 850° F. totaling 75% reduction. It is apparent that FIGURE 2B shows that the banding has been almost completely eliminated and that the structure is much more uniform, with good precipitate dispersion.

FIGURE 3 illustrates the marked effect of the preheat treatment of FIGURE 2B followed by additional hot working upon the earing characteristics of the fully fabricated sheet as compared to one heat treatment at the times and temperatures specified in curves **1** and **2**. It is apparent that the earing is considerably less with the re-heating at 1000° F. for six hours shown in curve **3**. Furthermore, the general tendency of reduced earing at lower ratios of (Mn+Fe):Si is also to be observed. The (Mn+Fe):Si ratio must be less than or equal to 7.5 to obtain earing values below 1%. By comparison with Curve **3** of FIGURE 1, it is believed that the low earing in curve **3** of FIGURE 3 is due to a smaller effective dendritic cell size and more uniform structure resulting from the re-heating and additional hot rolling.

FIGURE 4 shows the effect of the reheat treatment of FIGURE 2B followed by additional hot rolling upon the grain size of fully fabricated sheets. It is apparent that the grain size is considerably smaller (more grains per cu. mm) than with the single pre-heat treatment. Furthermore FIGURE 4 shows that when using rapid cooling rates of from 50–200° F./hr. after the preheat and rates of 50–300° F./hr. after the reheat fine grain sizes even in excess of 20,000 mm.^{3} can be obtained. These very fine grain sizes are to be contrasted with the best and much coarser value of 8736 grain mm.^{3} disclosed in U.S. Patent 3,219,491 which could only be obtained with very slow and uneconomical cooling rate of less than 50° F./hour.

The cooling rate in step 2 is very important. The cooling rate cannot be faster than 200° F. per hour, because if it is, the earing characteristics and grain size are adversely affected. It is believed that this is because manganese (and possibly iron) precipitation and growth will not take place if the cooling rate is greater than 200° F. per hour, but this is not certain.

However, the ratio must be at least 50° F. per hour or the time required to cool in shop processing becomes uneconomical. Not only the cost of fuel and other operational expenses for the furnace must be considered but also, the furnaces are tied up for long periods of time and cannot be used to process other material which must be shipped to customers on time.

In step 5, the cool from the reheat temperature of 950° F.—1150° F. at a rate of at least 50° F. per hour but not greater than 300° F. per hour again is very important. The rate must again be slow enough to insure additional precipitation and growth of the manganese (and possibly iron). On the other hand, the rate must be fast enough for economical commercial production. The cooling rate in this second cool can be somewhat faster than the first cool, however, because the original dendritic cell structure is largely broken up and diffusion distances for additional structural uniformity are considerably less.

Step 7 specifies a cool to room temperature which may be at any convenient rate, for example, at a rate of 50° F. per hour. However, it is not essential that this step take place.

If desired, the rolled product may be taken directly to step 8, which is an anneal at 550 to 800° F. for at least one minute and preferably for a time of from one to two hours. This post-hot-line-anneal has a very definite affect on both grain size and earing, as can be seen from Table I.

Table I shows the affect of this post-hot-line-anneal at 650° F. for 2 hours, prior to further cold rolling and annealing, on earing and grain size properties of material processed by our treatment. It is apparent that with an anneal at 650° F. for 2 hours, earing and grain size are substantially reduced in all cases.

TABLE I

Earing and grain size comparison when fabrication includes a post-hot-line-anneal at 650° F. for 2 hours. All data derived from commercially cast ingots containing 1.06% Mn, 0.57% Fe, 0.23% Si, 0.13% Cu.

Treatment History: 1150° F./16 hr. preheat, cooled at 75° F./hr., 30% reduction by hot rolling at 850° F., reheat to 1000°F./6 hrs., cool at 200° F./hr. Finish hot roll reduction at 850° F.

	Earing percent at 45°		Grain Size in gr./mm.^{3}	
	No anneal	Anneal (650° F./ 2 hr.)	No anneal	Anneal (650° F./ 2 hr.)
Gauge:				
.050"----------	1.9	1.3	4,630	8,300
.015"----------	1.2	0.8	3,300	5,000

The observed differences in earing and grain size are believed to result from a precipitation which takes place during the anneal. Furthermore, the grains which were elongated following the second hot rolling operation have recrystallized and are now equiaxed.

After the anneal, the product is cooled to room temperature, at any convenient rate, for example, at a rate of approximately 50–100° F. per hour.

The product is then cold rolled and the cold rolling has a considerable affect on grain size, as can be seen from FIGURE 4. It is apparent that the extent of prior cold rolling reduces the annealed grain size per se, and, furthermore, that the combination of the two-step hot rolling, interspersed with a reheat treatment at 950–1100° F., results in an even finer grain size. Grain size values of above 11,000 grains per cubic millimeter may be obtained with 70% reductions.

After this cold roll, there must be an anneal, which is to be carried out at 550–800° F. for a period of time of one minute to 24 hours, to ensure recrystallization.

If desired, two or more cycles of cold rolling and annealing may take place, depending on the desired finished gage and the grain size desired.

Also, for wrought alloys when combined with a low temperature precipitation treatment subsequent to the hot line fabrication, the process is exceptionally effective in grain size refinement.

The following examples illustrate the invention without limiting its scope.

EXAMPLE I

A single 3003 aluminum alloy was commercially cast using the direct chill method of casting. The ingot, consisting of 1.06% Mn, 0.57% Fe, 0.23% Si and 0.13% Cu was cast under plant conditions to a finished ingot cross section of 20" x 40". Casting drop rate was such that the dendritic cell size ranged from 25 to 40 microns in diameter.

A transverse slice from the ingot was reduced to 3" x 4" x 6" pieces. Two of the pieces were designated A and B and given the following treatment.

Piece A was preheat treated at 1050° F. for 25 hours prior to being hot rolled to sheet thickness. Piece B received an 1150° F./5 hour preheat treatment. Both were cooled at the approximate rate of 85° F. per hour to hot rolling temperature.

Hot rolling of each slice was done at a temperature of 750° F. ± 25° F.–50° F. to 0.500". The 0.500" plate was allowed to cool to 150–200° F. to simulate finish rolling and the plate rolled to 0.160" sheet. Upon cooling to room temperature the sheet was given a post-hot-line-coil anneal at 650±10° F. for 4 hours with a heat-up rate controlled at 50° F./hour to temperature. Subsequent to the post-hot-line-anneal, each sheet received a 70% nominal cold reduction in thickness and a final anneal at 650° F. for 4 hours. Again, the heat-up rate for the final anneal was controlled at 50° F./hour.

Earing and grain size results for the finished .050" (nominal) sheet are given below in Table II. It will be seen that these treatments (A and B) are ineffective in grain refining and earing reduction.

A third portion of the ingot (hereafter ingot C) was processed as follows. Ingot C received a preheat treatment identical to ingot B. However, following the initial 30% reduction in thickness during hot rolling, the slab was reheated to 1050° F. for 6 hours. Upon cooling at the rate of approximately 100° F. per hour to the original hot rolling temperature of 725° F. ± 25° F., hot rolling and further fabrication continued as indicated for ingots A and B.

The test results given in Table II show that the invention (Ingot C) was highly effective in reducing grain size and earing.

TABLE II.—EARING AND GRAIN SIZE RESULTS AT .050"-0 TEMPER

Ingot	Grain size (grns./mm.^{3})	Earing height (in percent)
A	4,700	3.3
B	5,000	2.2
C	9,500–13,000	0.8–1.0

EXAMPLE II

An ingot of 3003 aluminum alloy consisting of 1.16% Mn, 0.63% Fe, 0.19% Si, 0.15% Cu was commercially cast using the direct chill method of casting. The ingot had a cross section size of 19.5" by 40". The dendritic cell size ranged from 25 to 40 microns in diameter. The ingot was heated at 1125° F. for five hours. It was cooled to hot rolling temperature at a rate of 85° F. per hour and it was then hot rolled at a temperature of 850° F. to a reduction of 30%. It was then reheated to a temperature of 1018° F. and held for 6.4 hours. It was cooled to a second hot rolling temperature of 820° F. at a rate of 100° F. per hour and it was hot rolled to an 80% reduction. The so-rolled product was cooled to room temperature, annealed at 650° F. for one hour, cooled to room temperature, cold rolled 70% to .050" and then annealed at 650° F. for two hours. Following this treatment, the product had a grain size of approximately 7,500 grains per cubic millimeter and had an earing height percent of approximately 0.5%. This product was found to be satisfactory for deep drawing both from a standpoint of earing and grain size. A second cold rolling operation of 70% to .015" followed by an anneal yielded a product having a grain size of 12,500 gr./mm.^{3} and with earing height of 0.3%.

It is to be understood that the invention is not limited to the illustrations described and shown herein, which are deemed to be merely illustrative of the best modes of carrying out the invention, and which are susceptible to modifications of form, size, arrangement of parts and detail of operation. The invention rather is intended to encompass all such modifications which are within the spirit and scope of the invention, as set forth in the appended claims.

What is claimed is:

1. A process for treating cast aluminum alloys containing manganese in the range from about 0.5–2.0% by weight comprising:
   - (A) heating the ingots at a temperature of 950 to 1150° F. for 5 to 25 hours.
   - (B) cooling the ingots at a rate of between 50 to 200° F. per hour to a temperature of from 700 to 950° F.
   - (C) hot working the ingots from 10 to 70% within the temperature range of 700 to 950° F.
   - (D) reheating the worked ingots to 950 to 1100° F. and holding within this temperature range for 5 to 25 hours,
   - (E) cooling at a rate from 50 to 300° F. per hour to 700 to 950° F.,
   - (F) hot rolling at least 75% in the 700 to 950° F. temperature range,
   - (G) annealing at 550 to 800° F. for at least one minute,
   - (H) cooling to room temperature,
   - (I) cold rolling at least 10%, and then
   - (J) annealing the cold rolled product.

2. A process according to claim 1 in which the casting is done by the direct chill method.

3. A process according to claim 1 in which the aluminum alloy is 3003 aluminum.

4. A process according to claim 1 in which more than one cold rolling step, interspersed with an anneal, is carried out at the end of the process.

5. A process according to claim 1 in which low earing and a fine grain size are obtained in the product.

6. A process according to claim 3 in which cold rolling is carried out to a reduction of at least 70% and a grain size of at least as fine as 10,000 grains per cubic millimeter is obtained.

7. A process according to claim 5 in which cold rolling is carried out to a reduction of at least 70% and a grain size of at least as fine as 10,000 grains per cubic millimeter and earing of not greater than 1% is obtained.

8. A process according to claim 1 in which the as-cast dendritic cell size averages at least 25 microns.

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