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**DIE-EXPRESSED ARTICLE OF ALUMINUM-BASE ALLOY AND METHOD OF MAKING**

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The invention relates to die-expressed articles of aluminum-base alloy. It more particularly concerns an improved method of producing die-expressed articles of dispersion-hardened aluminum-base alloys whereby good mechanical properties are attained which are not appreciably lowered by exposure to elevated temperatures.

This application is a continuation-in-part of a prior-filed application, Serial No. 810,258 filed May 1, 1959, now abandoned.

Heretofore high strength aluminum-base alloys have been prepared by alloying aluminum with one or more suitable constituents which are capable of forming solid solutions with aluminum. Such alloys are often further treated, as by solution and precipitation heat treating and cold-working, to increase yield and tensile strength. However, at elevated temperatures the effectiveness of solid solution strengthening and precipitation hardening is limited and further the benefits of cold-working and aging are soon lost because recovery, recrystallization and overaging take place upon heating the alloys to even rather moderate temperatures. In other attempts to produce high strength aluminum-base alloys, fine aluminum powder having a thin oxide coating has been die-expressed to produce an extrude in which aluminum oxide is widely and finely dispersed in an aluminum metal matrix. While so-formed extrudes exhibit good mechanical properties, they tend to be quite brittle and difficult to form even at elevated temperatures. In addition, extremely fine aluminum powder is required to provide sufficiently wide dispersion of the oxide in the extrude as the oxide is present only as a surface coating on the so-particulated metal particles. The preparation of aluminum powder of sufficient fineness is not only expensive but the fine powder is hazardous to handle.

It is therefore an object of the invention to provide an improved method of forming an aluminum-base alloy having superior physical and mechanical properties at elevated temperatures.

A further object is to provide an improved method of preparing an aluminum-base alloy which has superior mechanical and physical properties but is still readily formable into useful shapes.

A still further object of the invention is to provide an improved aluminum-base alloy having superior mechanical and physical properties which are not appreciably decrease by exposure to elevated temperatures.

These and other objects and advantages of the invention will be more fully understood on becoming familiar with the following description and the appended claims.

This invention is predicated on the discovery that, by preparing an aluminum-base alloy containing aluminum and one or more constituents which are each miscible with aluminum in the molten state but substantially insoluble in solidified aluminum and by rapid solidification of said alloy, for example, into a mass of atomized particles, heating, compacting and then die-expressing the solidified metal to form an extrude or die-expressed article, the so-obtained article exhibits exceptionally desirable mechanical properties. For purposes of the specification and claims the term "solidified aluminum" is extended in meaning to include solidified aluminum-base alloy.

In carrying out the invention, an aluminum-base alloy

containing at least 70 percent of aluminum is prepared by alloying, according to well known methods, aluminum and one or more constituent metals having the hereinafter defined specific properties. An essential constituent metal must have sufficient solubility in molten aluminum at reasonable alloying temperatures, for example 650° to 900° C., to avoid the necessity of employing high temperatures at which aluminum readily attacks container materials, but a solid solubility in aluminum and aluminum-base alloy of less than 0.1 atomic percent. The use of metals, such as boron, which exhibit the requisite low solid solubility in solidified aluminum but which are difficult to alloy in effective amount in molten aluminum is undesirable in the practice of the invention. As to the solid solubility in aluminum, those elements which on cooling form an inter-metallic compound with aluminum, and which are insoluble in solid aluminum are employed. The insoluble phase must remain suspended in the rapidly cooled and solidified aluminum. It is further desirable that the molten alloy exhibit a narrow solidification range so that size of the intermetallic compound particles is minimized. It is desired that the intermetallic particles dispersed in the solid aluminum have a maximum diameter of about 0.0001 inch or less, preferably as small as about 0.00005 inch or less since the smaller the particle size the more the improvement in properties that results. A few particles having longer diameters can be tolerated but they should be kept to a minimum to avoid adversely affecting the properties of the final product. Suitable metal constituents that may be used singly or in combination in the practice of the invention include gold, barium, rare earth metals, palladium, platinum, antimony, selenium, strontium, tellurium, thorium and uranium, the pertinent properties of which are listed in Table I.

TABLE I

Metal constituent	Solid solubility in Al, Wt. percent	Liquid solubility in Al at 800° C., Wt. percent	Solidus temperature of Al Binary, ° C.	Inter-metallic compound formed with Al	Wt. percent of constituent to form 10% by volume of inter-metallic compound (estimated)
Au.....	1	30	642	AuAl <sub>2</sub> .....	20
Ba.....	nil	10	652	BaAl <sub>4</sub> .....	10
Ce.....	0.05	20	638	CeAl <sub>3</sub> .....	10
Pd.....	nil	50	615	PdAl <sub>3</sub> .....	8
Pt.....	nil	40	639	PtAl <sub>3</sub> .....	12
Sb.....	0.1	10	657	SbAl <sub>3</sub> .....	18
Se.....	nil	22	648	Se <sub>2</sub> Al <sub>3</sub> .....	15
Sr.....	nil	( <sup>1</sup> )	( <sup>1</sup> )	SrAl <sub>4</sub> .....	-----
Te.....	nil	53	621	TeAl <sub>3</sub> .....	20
Th.....	0.01	30	632	ThAl <sub>3</sub> .....	18
U.....	nil	20	640	UAl <sub>3</sub> .....	15

<sup>1</sup> Similar to barium.

The alloy to be used is brought to the molten state in any convenient manner in preparation for rapid solidification. Temperatures in the order of 25 to 50 centigrade degrees above the melting point of the alloy are desirable although other temperatures may be used at which the alloy is in the molten state. It is preferable to use the lower temperature of a molten state not only so as to reduce the degree of hazard involved in handling the molten alloy but also to reduce the amount of heat which must be removed to permit the molten alloy to return to the solid state. It is highly desirable for reasons hereinafter more fully discussed that the solidification of the atomized alloy take place quickly in order to minimize aggregation or crystal growth of intermetallic compounds which are insoluble in solidified aluminum.

The alloy while in the molten state may be subjected to a dispersion and chilling operation whereby the metal is obtained in atomized form, that is, in the form of fine

individually frozen discrete pellets. There are various ways in which atomization may be performed and any one of them may be used. A convenient method appears to be directing a jet of an inert cooling gas against an unconfined stream of the molten alloy as described in U.S. Patent No. 2,630,623.

For example, a freely falling stream of the molten metal may be broken into droplets and solidified by impinging upon the stream an inert gas such as a hydrocarbon gas (e.g., methane, ethane, propane, butane, etc.), argon, helium, hydrogen, the inert gas having a boiling temperature below the melting point of the molten metal. A wide range of pellet sizes, although small, usually results from the atomizing operation. The atomized product comprises more or less spherical pellets for the most part ranging in size from about 10 mesh to smaller than 325 mesh. A preferred range of pellet sizes is from about 325 mesh to about 140 mesh because of the outstanding properties achieved. However, very good properties are achieved with pellet sizes predominating in the 30 to 60 mesh range.

The amount of reduction in the cross-sectional dimensions of the compact effected by the extrusion or die-expression is subject to wide variations and may be from about 5 to 1 to as much as 200 to 1 or more (i.e., from about 80 percent to over 99 percent reduction in cross-sectional area).

The so-produced extrude having a uniform dispersion of finely-divided intermetallic compounds exhibits enhanced properties at both room and elevated temperatures and is less adversely affected by fabrication or heat treatment at high temperatures.

#### Example

In accordance with the invention, a quantity of each of the aluminum-base alloys listed in Table II in atomized form was provided. In each instance, the atomized pellets contained a uniform dispersion of intermetallic compound in which the intermediate compound had an average diameter of about 0.0005 inch, substantially none of the intermetallic compound having a diameter greater than 0.0001 inch.

TABLE II

Run No.	Composition				Properties at 24° C.				Properties at 316° C.			Properties at 427° C.		
	Per-cent Th	Per-cent Ba	Per-cent MM	Per-cent Al	Per-cent E	TYS	CYS	TS	Per-cent E	TYS	TS	Per-cent E	TYS	TS
1-----	12.5	-----	-----	bal.	16	24	24	38	31	10.5	13.3	35	4.0	6.3
2-----	18.1	-----	-----	bal.	15	26	25	48	21	9.0	12.4	32	3.6	5.4
3-----	-----	-----	11.5	bal.	17	22	24	41	-----	8.7	-----	16	3.7	5.5
4-----	-----	9.1	-----	bal.	12	20	20	33	14	8.8	11.8	14	4.1	5.8

MM=Misch Metal.

Percent E=Percent elongation in 2 inches.

TYS=Tensile yield strength in thousands of lbs. per sq. in.

CYS=Compression yield strength in thousands of lbs. per sq. in.

TS=Ultimate tensile strength in thousands of lbs. per sq. in.

These pellets exhibit very desirable mechanical properties and may be used as pellets per se, for example, to reinforce other metals, as a load support, etc. or may be further fabricated, as by extrusion, rolling, and the like.

Of course, other methods of rapidly quenching appropriate alloy compositions, as well as other methods of atomizing aluminum may be employed.

As a result of the atomizing operation, there is imparted to each pellet of the aluminum-base alloy a special heterogeneous microstructure essential in achieving the objects of this invention. This structure is characterized by an aluminum metal matrix having uniformly dispersed therethrough a discontinuous phase made up of very fine crystallites of an intermetallic compound thereof with aluminum. The intermetallic compound is present in an amount totalling, by volume, from 0.5 to 20 percent of the alloy and preferably, from 5 to 15 percent.

In the next step of the method, the rapidly solidified metal is heated in preparation for compacting and die-expression. Compacting and die-expression may be carried out in conventional apparatus designed for the extrusion of aluminum-base alloy. A suitable method and apparatus for carrying out the die-expression of pelletized light metal, such as aluminum, is described in U.S. Patent No. 2,630,623. The temperature to which the metal is heated is within the conventional plastic deformation temperature range for aluminum-base alloys, usually between about 250° C. and 500° C. but always below that temperature which affects the dispersion through agglomeration.

It has been found that the as-atomized aluminum-base alloy may be heated in bulk to the desired temperature merely by placing it in a suitable metal vessel in a heated oven. Or it is possible to charge the heated container of a die-expressing apparatus with as-atomized metal and proceed with the operation of the apparatus to effect die-expression with substantially no destruction of the as-atomized microstructure of the alloy.

In another embodiment of the invention, an aluminum-base alloy is prepared by making suitable additions to aluminum of a metal constituent, which forms an insoluble phase therewith as described hereinabove as well as one or more metals which increase the strength of aluminum in a well known conventional manner such as by solution hardening. It has thus been found that the benefits of dispersion hardening may be combined with the benefits of increasing the strength of the matrix about the finely dispersed crystallites of solid insoluble phase. Metals which may be added variously to increase the matrix strength include:

	Weight percent
Ag -----	0-10
Ca -----	0-0.7
Cr -----	0-0.8
Cu -----	0-6
Li -----	0-5.5
Mg -----	0-15
Mn -----	0-2
Si -----	0-2
Ti -----	0-1
Zn -----	0-10
Zr -----	0-0.3

Use of combinations of the above-listed elements in amount in which the metals are incompatible by virtue of mutual insolubility in molten aluminum whereby a precipitate is formed which settles from the melt, or in amounts which otherwise fail to increase matrix strength, is of no advantage in the practice of the invention. While simple binary or ternary combinations of the above-listed matrix strengthening constituents are believed to be compatible, it is within the skill of the metallurgist to check desired alloying combinations for incompatibility such as insolubility in molten aluminum. In general, any conventional aluminum-base alloy system may be employed, such as Al-Mg, Al-Cu, Al-Mn, Al-Si, Al-Zn,

Al—Mg—Zn, or Al—Si—Cu, in combination with one or more of the previously mentioned metals which form a solid insoluble phase in solidified aluminum.

Such composite alloys, that is, those including dispersion hardening, as well as conventional strengthening alloying metals, are atomized, compacted, and extruded as described hereinabove.

Various modifications may be made in the present invention without departing from the spirit or scope thereof and it is to be understood that I limit myself only as defined in the appended claims.

What is claimed is:

1. A die-expressed article of aluminum-base alloy comprising at least about 70% aluminum and from 0.5 to 20% by volume of particles of solid insoluble intermetallic compound intimately and uniformly dispersed throughout said die-expressed article, said solid insoluble intermetallic compound being selected from the group consisting of AuAl<sub>2</sub>, BaAl<sub>4</sub>, CeAl<sub>4</sub>, PdAl<sub>3</sub>, PtAl<sub>3</sub>, SbAl, Se<sub>3</sub>Al<sub>2</sub>, SrAl<sub>4</sub>, TeAl<sub>3</sub>, ThAl<sub>3</sub>, UAl<sub>4</sub>, and mixtures thereof and said particles having a maximum diameter of about .0001 inch.

2. A die-expressed article of aluminum-base alloy comprising at least about 70% aluminum and from 0.5 to 20% by volume of solid insoluble AuAl<sub>2</sub> particles intimately and uniformly dispersed throughout said die-expressed article, said AuAl<sub>2</sub> particles having a maximum diameter of .0001 inch.

3. A die-expressed article of aluminum-base alloy comprising at least about 70% aluminum and from 0.5 to 20% by volume of particles of solid insoluble ThAl<sub>3</sub> intimately and uniformly dispersed throughout said die-expressed article, said ThAl<sub>3</sub> particles having a maximum diameter of 0.0001 inch.

4. A die-expressed article of aluminum-base alloy as in claim 1 in which the volume of particles of solid insoluble intermetallic compound is in the range of 5 to 15%, said particles having a maximum diameter of about 0.00005 inch.

5. A die-expressed article of aluminum-base alloy consisting essentially of from 0.5 to 20% by volume of solid insoluble particles of aluminum intermetallic compound and the balance aluminum-base alloy containing at least one matrix strengthening alloying constituent selected from the group consisting of silver, calcium, chromium, copper, lithium, magnesium, manganese, silicon, titanium and zinc; said particles of aluminum intermetallic compound being selected from the group consisting of AuAl<sub>2</sub>, BaAl<sub>4</sub>, CeAl<sub>4</sub>, PdAl<sub>3</sub>, PtAl<sub>3</sub>, SbAl, Se<sub>3</sub>Al<sub>2</sub>, SrAl<sub>4</sub>, TeAl<sub>3</sub>, ThAl<sub>3</sub>, UAl<sub>4</sub> and mixtures thereof; said particles of aluminum intermetallic compound being intimately and uniformly dispersed throughout said aluminum-base alloy; said particles of aluminum intermetallic compound having a maximum diameter of 0.0001 inch; and said die-expressed article comprising at least 70% of aluminum.

6. The method of making a die-expressed article of an aluminum-base alloy comprising at least 70% by weight of aluminum and from 0.5 to 20% by volume of particles of solid insoluble intermetallic compound selected from the group consisting of AuAl<sub>2</sub>, BaAl<sub>4</sub>, CeAl<sub>4</sub>, PdAl<sub>3</sub>, PtAl<sub>3</sub>, SbAl, Se<sub>3</sub>Al<sub>2</sub>, SrAl<sub>4</sub>, TeAl<sub>3</sub>, ThAl<sub>3</sub>, UAl<sub>4</sub>, and mixtures thereof, said particles being uniformly and intimately dispersed through said aluminum, which comprises: rapidly solidifying the requisite alloy from the molten state so as to form and disperse said intermetallic compound particles throughout said aluminum with a maximum particle size of .0001 inch; compacting and extruding the resulting aluminum having the fine crystallites dispersed therethrough thereby to provide a die-expressed article.

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