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(54) **ALLOY TOUGHENING METHOD.**

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(56) References cited:
EP-A- 0 136 508
US-A- 3 899 319
US-A- 4 365 994
GB-A- 2 088 409
US-A- 4 347 076
US-A- 4 379 719

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PROCEEDINGS OF THE 43RD ANNUAL MEETING ON THE ELECTRON MICROSCOPY SOCIETY OF AMERICA, 1985, pages 32,33, G.W. BAILEY Ed., San Francisco Press, Inc., San Francisco, CA, US; M.G. CHU et al.: "Microstructural evolution during solidification of Al-Fe-Ce powders"

MATERIALS RESEARCH SOCIETY SYMPOSIUM RPOCEEDINGS, vol. 28, 1984, pages 21-27, Elsevier Science Publishing Co., Inc., Amsterdam, NL; V. LAXMANAN: "Some fundamental considerations during rapid solidification processing"

Materials Science and Engineering Vol. 65 (1984), H.Jones "Microstructure of Rapidly Solidified Materials", pp.145-156, esp. p.145&146

Properties related to Fracture Toughness, ASTM STP 605,1976 J.T. Staley "Microstructure and Toughness of High Strength Aluminum Alloys ", pp. 71-103, esp. p.80 & 82.

Scripta Metallurgical vol. 18, 1984 D.J. Skinner et al, " High Strength Al-Fe-V Alloys at Elevated Temperatures Produced by Rapid Ouenching from the Melt ", pp. 905 - 909

Description

Metallurgical objects produced from rapidly cooled metal have been burdened by low toughness. The cause of this low toughness was not known.

It is an object of the invention to provide a method for toughening metallurgical objects produced from rapidly cooled metal components.

We have discovered that metastable, featureless regions in rapidly cooled metal adversely affect toughness.

According to the present invention, there is provided a method of treating a metallurgical object or metal particles to improve toughness of the object or object formed by bonding the particles together wherein the object or the particles contain metastable featureless regions adversely, affecting the toughness of the object or object formed from the particles, comprising heating the object or the particles for transforming the regions at least sufficiently out of their metastable state to stabilize them and make them deformable, and deforming the object or object formed from the particles to improve the toughness of the object or object formed from the particles. Preferred embodiments are defined in the dependent claims 2 to 9.

Figure 1 , composed of Figures 1a to 1d, are photomicrographs of a powder used in the invention.

Figures 2 to 4 are plots of data.

Featureless Regions

The present invention concerns a treatment of metallurgical objects containing certain metastable, featureless regions. The treatment improves fracture toughness.

Instances in the literature where the term "featureless" is used to refer to these regions are as follows:

Location in Reference	Citation of Reference
Col. 4, line 21	U.S. Pat. No. 3,899,820, 8/19/85
E.g. lines 7&8, abstract	RapidlyQu'dMetalsIII,1,73-84,1978
E.g., the title	Met.Trans.A,V.15A,1/84,pp29-31
Intro.,2nd.para.,line2	Scrip.Met'ica,V18,1984,pp905-9
Intro.,2nd.para.,line6	Scrip.Met'ica,V18,1984,pp911-6
E.g., page 26	MatResSocSympProc,V28,1984,pp21-7
Pg. 148, top left col.	Mat.Sci.&Eng.,V65,1984,pp145-56
3rd.para.,line2	43rdAnMt'gElecM'scopSoc,'85,pp32-3
Pg.9, line 6	EP-A-0 136 508, 4/10/85

These featureless regions are crystalline. This is evident alone in the title of the second-listed reference, "Rapidly Quenched Crystalline Alloys". It is also evident from what is believed to be the pioneer article on these regions, entitled "Observations on a Structural Transition in Aluminum Alloys Hardened by Rapid Solidification" by H. Jones, Mater.Sci.Eng., 5 (1969/70),pp. 1-18. Thus, in the Summary of the article by Jones, refrence is to X-ray diffraction alpha-Al line broadening, and shift, in zone A regions ("zone A regions" is synonymous to "featureless regions", as can be observed, for instance, in the references antedating Jones, as cited in the preceding paragraph), such indicating that discussion is of crystalline material.

The ninth-listed reference, EP-A-0 136 508, discloses an aluminum-based alloy having high strength at elevated temperatures which may be comminuted and processed into articles by deformation at high temperatures. It further discloses a method and apparatus for forming rapidly solidified metal having a desired microstructure. In alloys cast by employing the apparatus and method, optical microscopy reveals a uniform, featureless morphology.

The featureless regions result from rapid cooling. Figure 1 illustrates the phenomenon of featureless regions. In Figure 1a, taken using optical microscopy, the featureless regions appear white as compared to the other regions which have a texture that appears to be black specks on a gray background. Note that the smaller particles tend to be completely featureless, an effect of the higher cooling rate experienced by the smaller particles. The scanning electron microscopy photographs of Figures 1b-1d further illustrate the featureless regions, which appear uniformly gray as compared to the remaining, dendritically textured regions. Figures 1b and 1d show again the smaller, completely featureless regions. Figure 1c shows in particularly good detail that the particle has a featureless half-moon region on its lower side. This is an aspect which also shows in Figures 1a and 1b, namely that higher cooling rates in some parts of a particle versus slower cooling rates in other parts can lead to a situation where the particle will be featureless in the rapidly cooled parts and textured in the slower cooled parts.

Alloys

In general, any alloy containing featureless regions can be treated according to the invention.

A preferred Al alloy consists essentially of 4 to 12% Fe, 2 to 14% Ce, remainder Al. Fe combines with Al to form intermetallic dispersoids and precipitates providing strength at room temperature and elevated temperature. Ce combines with Fe and Al to form intermetallic dispersoids which provide strength, thermal stability and corrosion resistance. Further information concerning this alloy is contained in U.S. Patent Nos. 4,379,719 and 4,464,199.

Uniformizing

With respect to strength, such as yield or tensile strength, our uniformizing heat treatment, within the featureless regions, represents an overaging.

This heating step of the invention for the above preferred Al alloy will generally be in the range 750-950°F for 10 seconds to 4 hours. However, at lower temperatures, longer time may be suitable. This could be of advantage in the case of large billets, in order to obtain temperature uniformity.

Fast heating appears to be best (via induction heating), since this will prevent coarsening, for instance dispersoid coarsening.

Deformation

In the heating to effect the uniformizing of the invention, the featureless particles are stabilized and they become deformable. Deformation after the uniformizing treatment, for instance deformation in the form of compaction, extrusion or rolling, will provide a more uniform microstructure, with improved bonding between powder particles. Improved interparticle powder bonding further increases toughness and resistance to crack propagation.

Illustration

The following Table A illustrates results achieved by procedure according to the present invention (with heat treatment, i.e. 1 to 3 minutes at 900°F followed by cooling to 725°F extrusion temperature) compared to results without heat treatment (i.e. the billet was heated directly to the 725°F extrusion temperature and then extruded). Processing in going from extruded bar to sheet was the same in both instances.

TABLE A

Comparative Examples

	With Heat Treatment[a]		Without Heat Treatment	
	Toughness[b]	Strength[b]	Toughness[b]	Strength[b]
Extrusions	21.4	50.9	13.7	55.1
Sheet	720[c]	70.2	405[c]	73.7

[a] 1 min at 900°F

[b] Toughness = $Ksi \cdot in^{1/2}$, Strength = Ksi

[c] Sheet toughness given in unit propagation energy (UPE) $in\text{-}lb/in^2$

In the case of the extrusion, there was a 56% increase in toughness for an 8% decrease in yield strength. For the sheet, toughness was increased 78% for an 5% decrease in yield strength.

Advantages

The invention improves toughness and thermal stability in metallurgical objects based on rapid solidification processes. It is expected that creep behavior will also be improved.

Further illustrative of the invention are the following examples.

Example I

Rapidly solidified aluminum alloy powder of composition 8.4% Fe, 4.0% Ce, rest essentially aluminum, had featureless regions resulting from rapid cooling during formation of the powder. To make the powder, a pot of such composition was alloyed by adding high purity alloying elements to high purity aluminum. The melt was passed through a filter and atomized using high temperature flue gas to minimize the oxidation of the alloying elements. During atomization, the powder was continuously passed through a cyclone to separate the particles from the high velocity air stream. The majority of powder particles had diameters between 5 and 40 micrometers. Powder was screened to retain only less than 74 micrometers size powder and fed directly into a drum. Besides Fe, Ce, and Al, the powder had the following percentages of impurities: Si 0.14, Cu 0.02, Mn 0.04, Cr 0.01, Ni 0.02, Zn 0.02, Ti 0.01. The powder was found to have featureless regions in about the same quantity and distribution as shown in Figure 1. The particle size distribution of the powder was 4.4% in the range 44 to 74 micrometers and 95.4% smaller than 44 micrometers. Average particle diameter was 15.5 microns as determined on a Fisher Subsieve Sizer.

Billet was made from this powder by cold isostatic pressing to approximately 75% of theoretical density. Each 66 kg (145 lb) cold isostatic compact was encapsulated in an aluminum container with an evacuation tube on one end. The canned compacts were placed in a 658 K (725° F) furnace and continuously degassed for six hours, attaining a vacuum level below 40 microns. Degassed and sealed compacts were then hot pressed at 725° F to 100 percent density using an average pressure of 469.2 MPa (68 ksi).

A cylindrical extrusion charge measuring 15 cm (6.125 in.) diameter x 30.5 cm (12 in.) length was machined from the billet and subjected to a uniformizing treatments of 1 minute at 850° F and 1 minute at 900° F. Heating was done using an induction furnace operating at 60 H_z. Temperature was measured by a thermocouple placed at an axial location about 1.2 cm (0.5 in.) from the end. It took about 10 minutes to heat the extrusion charge from room temperature to 850° F or 900° F at which point temperature was controlled at 850° F and 900° F for the 1 minute holding time.

The extrusion charge was then air-cooled to 725° F and extruded as a bar of 5 cm (2 inches) x 10 cm (4 inches) cross section.

Another Al-Fe-Ce alloy having the composition Al-8.4%Fe-7.0%Ce was also uniformized at 900° F for 1 min.

Properties for both alloys are recorded in Table I. Results from Table I are shown graphically in Figure 2 . Note the strength toughness relation for the two different alloys.

TABLE I

Room Temperature Tensile and Fracture Toughness Test Results of Extrusions

Sample No.[a]	Alloy	Uniformizing Treatment Temp. °F	Uniformizing Treatment Time Min.	Yield Strength 0.2% Offset MPa	Yield Strength 0.2% Offset (Ksi)	Tensile Strength MPa	Tensile Strength (Ksi)	Elongation (%)	Fracture Toughness $MPa\cdot m^{\frac{1}{2}}$	Fracture Toughness $(Ksi\cdot in^{\frac{1}{2}})$
514295-1B	Al-8.4Fe-4.0Ce	Control		388	(56.2)	497	(72.0)	12.5	14.7	(13.4)
514282-1	Al-8.4Fe-4.0Ce	Control		380	(55.1)	469	(68.0)	9.6	15.1	(13.7)
514412-T	Al-8.4Fe-4.0Ce	850	1	366	(53.0)	449	(65.0)	17.8	19.6	(17.8)
514413-1B	Al-8.4Fe-4.0Ce	900	1	351	(50.9)	425	(61.6)	16.7	23.5	(21.4)
514398-2T	Al-8.4Fe-7.0Ce	Control		426	(61.7)	530	(76.8)	11.0	9.35	(8.5) [c]
514416-2T	Al-8.4Fe-7.0Ce	900	1	373	(54)	450	(65.2)	16.0	27.8	(25.3)

NOTES:

Values are averages from duplicate tests. Yield and tensile strengths were measured in the longitudinal (L) direction using 0.907 cm (0.357") diameter specimens machined from the extruded product. Elongation was measured in a 3.56 cm (1.40") gauge length. Tensile properties were obtained according to ASTM B557. Fracture toughness was measured in the L-T orientation using compact tension specimens of size 1.90 cm (0.75") thick x 3.81 cm (1.50 m) x 4.57 cm (1.80").

a) Product size: 5.1 cm x 10.2 cm (2.0 in. x 4.0 in.)

b) Values are Kic per ASTM E399.

c) This value was not a valid Kic but a meaningful value per ASTM B645

Example II

Extruded bar of Example I was rolled at 600°F to sheet of final thickness equalling 1.60 mm (0.063 inch).

Prior to rolling, the extrusion was sawed to approximately 25 cm (10 in.) lengths. Surface roughness, caused by pickup of aluminum on the extrusion dies, was eliminated by machining the extrusions to the thicknesses listed in Table III. Also listed are process parameters used to roll the Al-Fe-Ce 1.60 mm (0.063 in.) sheet.

Each piece was cross rolled until the desired width, greater than 41 cm (16 inches) was obtained, followed by straight rolling to the desired thickness, 1.60 mm (0.063 inch).

1.27 cm (0.5 in.) width x 5.08 cm (2.0 in.) gage length tensile specimens were prepared and tested to give results as shown in Table II. Sheet tensile strength was determined per ASTM E8 and E23. The Alcoa-Kahn tear test (see "Fracture Characteristics of Aluminum Alloys," J. G. Kaufman, Marshall Holt, Alcoa Research Laboratories, Technical Paper No. 18, pp. 10-18, 1965) and fracture toughness K_c per ASTM B646 and E561 were used to compare sheet toughness. These results are shown in Table II. Figure 3 shows the graphic representation of the strength/fracture toughness, K_c, relationships for representative samples of Table II, while Figure 4 provides a corresponding presentation from Table II in the form of toughness indicator, or unit propagation energy, against yield strength. The superiority of sheet treated according to the present invention compared to the ingot metallurgy representatives is apparent.

It is to be noted that for a given alloy, the tradeoff between strength loss and toughness improvement is a function of time and temperature during the uniformizing treatment.

TABLE II

Room Temperature Tensile and Fracture Toughness
1.60mm (0.063 in.) Sheet

Sample No.[a]	Alloy	Treatment Temp °F	Treatment Time Min.	Yield Strength MPa	Yield Strength Ksi	Tensile Strength MPa	Tensile Strength Ksi	Elongation %	Tear Test kJ/m²	Tear Test in.-lb/in.²	Fracture Toughness, kc MPa√m	Fracture Toughness, kc Ksi√in.	Valid[h]
514295-2B	Al-8.3Fe-4.0Ce	Control		508	73.7	546	79.1	6.8	70.9	405[b]	122.7	111.7	Yes
554314	Al-8.3Fe-4.0Ce	Control		523	75.8	575	83.4	10.0	68.9	395			
514388-2	Al-8.3Fe-4.0Ce	Control		524	76.0	561	81.3	6.5	69.2	395[f]			
514412-BR	Al-8.3Fe-4.0Ce	850	10	477	69.2	513	74.3	5.8	125.6	715[c]	180.8	164.5	No
514413-1BR	Al-8.3Fe-4.0Ce	900	1	484	70.2	518	75.1	6.0	125.7	720[d]	191.2	174.0	No
514408-2BR	Al-8.3Fe-4.0Ce	900	10	424	61.6	460	66.7	8.0	135.5	775	168.1	153.0	No
554311	Al-8.3Fe-4.0Ce	850	60	432	62.6	483	70.0	10.0	135.5	775	214.5	195.0	No
514398-2T	Al-8.4Fe-7.0Ce	Control		579	84.1	622	90.2	6.5	0	0[g]			
514416-2TR	Al-8.4Fe-7.0Ce	900	1	519	75.4	549	79.6	8.2	117.3	670[e]	98.9	90.0	Yes
	7075-T6	-	-	517	74.9	568	82.3	11.2	50.7	290	70.8	64.4	Yes
	7075-T73	-	-	416	60.3	494	71.6	10.6	89.2	510	-	-	
	2024-T81	-	-	482	69.8	512	74.2	6.6	29.7	170	-	-	
	2024-T6	-	-	367	53.2	464	67.2	9.2	48.1	275	-	-	

NOTES:

a) All tests were done in the L-T orientation. Sheet thickness varies from 1.60 to 1.78 mm (0.063" to 0.070") except 554311 which has a nominal thickness of 1.42 mm (0.056"). Al-Fe-Ce tensile and tear test results are averages of duplicate tests, Kc results are single tests. 7075 and 2024 results are averages of 2-10 tests.

b) One of the duplicates underwent rapid & diagonal fracture (UPE may be estimated and slightly high; included in average).

c) Both tests: diagonal fracture (tear strength and UPE may be slightly high; included in average).

d) One of the duplicates underwent diagonal fracture (tear strength and UPE may be slightly high; included in average).

e) One of the duplicates underwent rapid fracture (UPE was estimated, but not included in average shown).

f) One test: rapid and diagonal fracture - curve not reliable (energy near zero; not included in average shown).

g) Crack growth was unstable.

h) Invalidities are due to specimen size, i.e., specimen was not large enough to provide enough recoverable elastic energy to produce unstable crack growth in an elastic-stress field.

Specimen Sizes:

Tensile: Sheet thickness x 1.27 cm (0.5") wide specimen. Elongation was measured in 5.08 cm (2.0") gauge length.

Tear Test: Kahn-type, sheet thickness x 3.65 cm (1.44") x 5.72 cm (2.25").

Fracture Toughness: Center-crack, sheet thickness x 40.6 cm (16.0") x 111.8 cm (44.0").

TABLE III

Process Parameters Used To Roll 1.60mm (0.063 in.) Al-Fe-Ce Sheet

Sample No.	Rolling Temperature K	Rolling Temperature F	Extrusion Thickness cm	Extrusion Thickness in.	Sheet Thickness mm	Sheet Thickness in.
514295-2B	589	600	4.72	1.86	1.59	0.0625
554314	616/589	650/600*	4.45	1.75	1.55	0.061
514388-2	589	600	2.51	0.988	1.65	0.065
514412-BR	589	600	5.08	2.0	1.68	0.066
514413-1BR	589	600	5.08	2.0	1.69	0.0665
514408-2BR	589	600	5.08	2.0	1.70	0.067
554311	616/589	650/600*	4.45	1.75	1.37	0.054
514398-2T	589	600	4.65	1.83	1.54	0.0605
514416-2TR	589	600	4.76	1.875	1.60	0.063

* Extrusions were heated to 616°K (650°F) for the first rolling reductions and 589°K (600°F) for subsequent reductions.

Unless noted otherwise, percentages herein are on a weight basis.

Claims

1. A method of treating a metallurgical object or metal particles to improve toughness of the object or toughness in an object formed by bonding the particles together, wherein said object or said particles contain metastable, featureless regions adversely affecting said toughness of the object or object formed from the particles, characterized by comprising heating said object or said particles for transforming the regions at least sufficiently out of their metastable state to stabilize them and make them deformable, and deforming the object or object formed from the particles to improve said toughness of the object or object formed from the particles.

2. A method as claimed in claim 1, characterized by the heating being sufficient to provide at least a 10% improvement in toughness.

3. A method as claimed in claim 1 or 2, characterized by the object or particles comprising an aluminum alloy.

4. A method as claimed in claim 3, characterized by the object comprising an aluminum alloy of the class referred to as non-heat treatable or dispersion hardened.

5. A method as claimed in claim 3, characterized by the particle comprising an aluminum alloy of the class referred to as non-heat treatable.

6. A method as claimed in claim 4 or 5, characterized by the object or particle comprising bonded powder.

7. A method as claimed in claim 6, characterized by the object or particle comprising a dispersion hardened, bonded powder.

8. A method as claimed in claim 7, characterized by the alloy consisting essentially of 4 to 12% iron, 1 to 8% rare earth metal, balance aluminum.

9. A method as claimed in claim 8, characterized by the alloy consisting essentially of 6 to 10% iron, 2 to 6% cerium, balance aluminum.

Patentansprüche

1. Verfahren zum Behandeln eines metallurgischen Gegenstandes oder von Metallteilchen zum Verbessern der Zähigkeit des Gegenstandes oder der Zähigkeit in einem durch Binden der Teilchen aneinander gebildeten Gegenstand, wobei der Gegenstand oder die Teilchen metastabile, merkmallose Bereiche enthält bzw. enthalten, die die Zähigkeit des Gegenstandes bzw. des aus den Teilchen gebildeten Gegenstandes beeinträchtigen, dadurch gekennzeichnet, daß durch Erhitzen des Gegenstandes oder der Teilchen die Bereiche wenigstens so weitgehend aus ihrem metastabilen Zustand transformiert werden, daß sie stabilisiert und verformbar werden, und daß der Gegenstand bzw. der von den Teilchen gebildete Gegenstand verformt wird und dadurch die Zähigkeit des Gegenstandes oder des von den Teilchen gebildeten Gegenstandes verbessert wird.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Erhitzung so weitgehend ist, daß die Zähigkeit um mindestens 10% erhöht wird.

3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß der Gegenstand oder die Teilchen wenigstens teilweise aus einer Aluminiumlegierung bestehen.

4. Verfahren nach Anspruch 3, dadurch gekennzeichnet, daß der Gegenstand wenigstens teilweise aus einer Aluminiumlegierung der als unvergütbar oder dispersionsgehärtet bezeichneten Klasse besteht.

5. Verfahren nach Anspruch 3, dadurch gekennzeichnet, daß das Teilchen wenigstens teilweise aus einer Aluminiumlegierung der als unvergütbar bezeichneten Klasse besteht.

6. Verfahren nach Anspruch 4 oder 5, dadurch gekennzeichnet, daß der Gegenstand oder das Teilchen wenigstens teilweise aus gebundenem Pulver besteht.

7. Verfahren nach Anspruch 6, dadurch gekennzeichnet, daß der Gegenstand oder das Teilchen wenigstens teilweise aus einem dispersionsgehärteten gebundenen Pulver besteht.

8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, daß die Legierung im wesentlichen aus 4 bis 12% Eisen, 1 bis 8% Seltenerdmetall, Rest Aluminium besteht.

9. Verfahren nach Anspruch 8, dadurch gekennzeichnet, daß die Legierung im wesentlichen aus 6 bis 10% Eisen, 2 bis 6% Cerium, Rest Aluminium besteht.

Revendications

1. Un procédé de traitement d'un objet métallurgique ou de particules métalliques pour améliorer la ténacité de l'ojet ou d'un objet formé en liant les particules ensemble, selon lequel l'objet ou les particules contiennent des régions métastables sans particularité ayant une influence défavorable sur la ténacité de l'objet ou d'un objet formé à partir des particules, caractéris en ce qu'il comprend le chauffage de l'objet ou des particules pour transformer les régions au moins suffisamment à l'extérieur de leur état métastable pour les stabiliser et les rendre déformables et la déformation de l'objet ou d'un objet formé à partir des particules pour améliorer la ténacité de l'objet ou d'un objet formé à partir des particules.

2. Un procédé selon la revendication 1, caractérisé en ce que le chauffage est suffisant pour produire une amélioration de la ténacité d'au moins 10%.

3. Un procédé selon la revendication 1 ou 2, caractérisé en ce que l'objet ou les particules comprennent un alliage d'aluminium.

4. Un procédé selon la revendication 3, caractérisé en ce que l'objet comprend un alliage d'aluminium de la classe désignée comme non apte au traitement thermique ou durci par dispersion.

5. Un procédé selon la revendication 3, caractérisé en ce que les particules comprennent un alliage d'aluminium de la classe dénommée non apte au traitement thermique.

6. Un procédé selon la revendication 4 ou 5, caractérisé en ce que l'objet ou les particules comprennent une poudre liée.

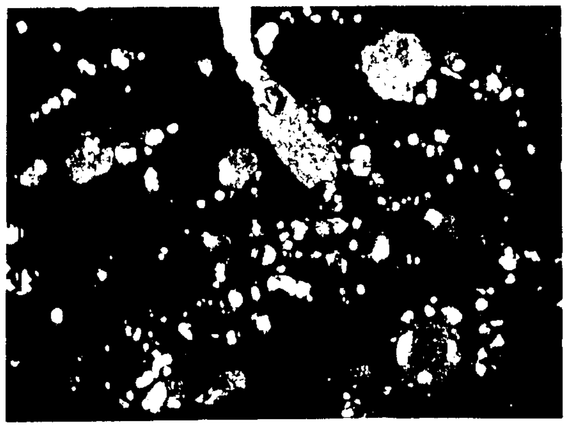
7. Un procédé selon la revendication 6, caractérisé en ce que l'objet ou les particules comprennent une poudre liée, durcie par dispersion.

8. Un procédé selon la revendication 7, caractérisé en ce que l'alliage consiste essentiellement en 4 à 12 % de fer, 1 à 8 % de métal des terres rares et le reste d'aluminium.

9. Un procédé selon la revendication 8, caractérisé en ce que l'alliage consiste essentiellement en 6 à 10 % de fer, 2 à 6 % de cérium et le reste d'aluminium.

FIG. 1: As Atomized Powder S No. 514283
(Al-7% Fe-6.2% Ce)

FIG.1a



40 μm

FIG.1b

2.5 μm

Optical

SEM

FIG.1c

3.6 μm

FIG.1d

7.4 μm

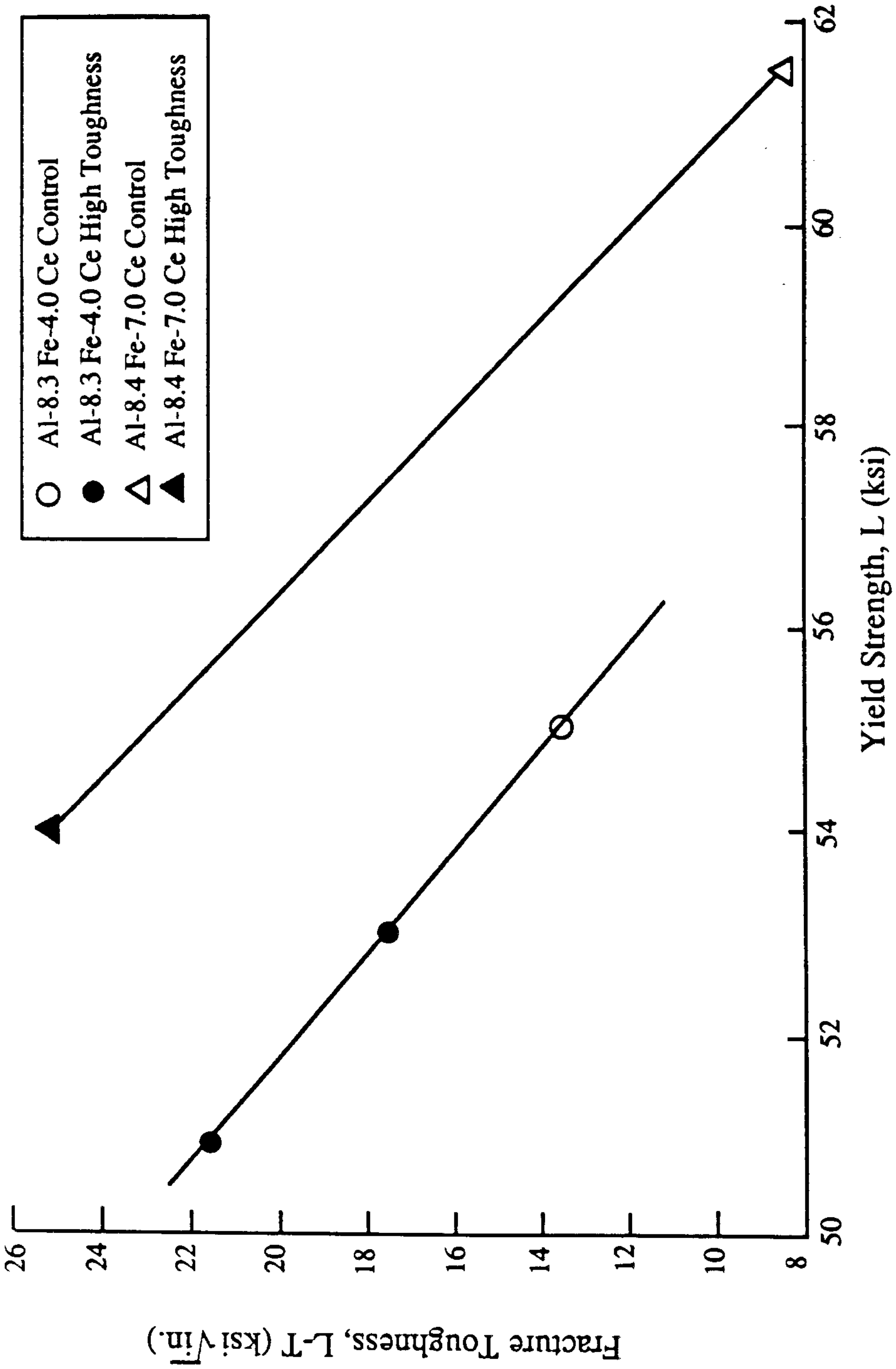


Figure 2: Fracture Toughness as a Function of Yield Strength 5.1 X 10.2 cm (2.0 X 4.0 in.) Extrusions

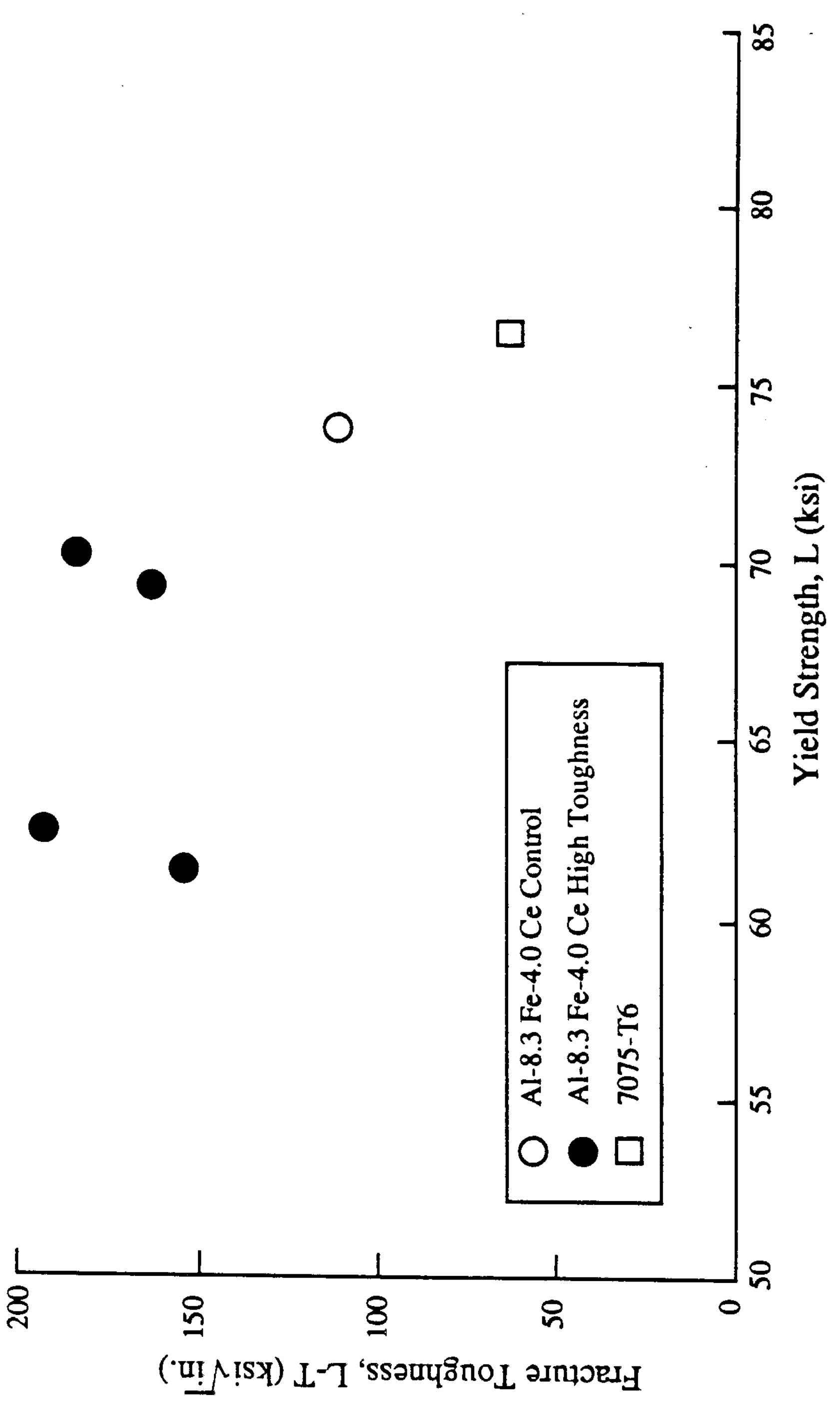


Figure 3: Fracture Toughness as a Function of Yield Strength 1.60 mm (0.063 in.) Sheet

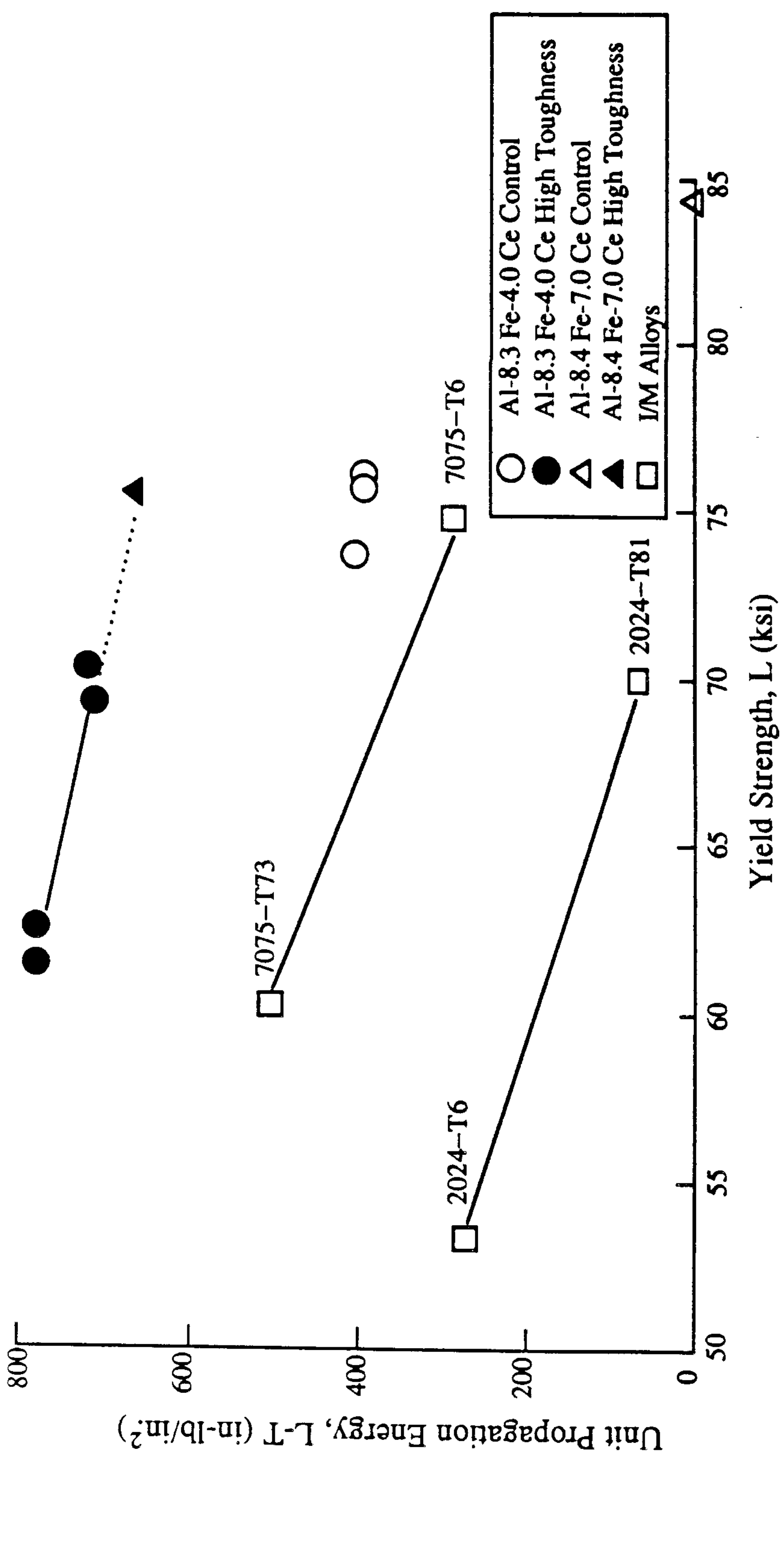


Figure 4: Unit Propagation Energy as a Function of Yield Strength 1.60 mm (0.063 in.) Sheet