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Elevated Temperature Strength and Microstructure of Atomized and Ball-milled Al-xFe-yCr Alloys

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Abstract Gas atomization, mechanical alloying and hot pressing have successfully made high temperature Al-9.45Fe-4.45Cr alloy. The microstructure and mechanical properties of this alloy has been studied by using optical microscope, scanning electron microscope, transmission electron microscope, X-ray diffractometer and compressive tester. It contains high concentration of transition elements of Fe and Cr, which form thermally stable dispersoids in the aluminum matrix. Proper oxidation of powders during ball milling strengthens the bulk extrudates by providing the obstacle particles. The oxide particles are very chemically and thermally stable and prevent the coarsening of the intermediate compounds.

1. Introduction

As the demands of an aerospace and automobile industries for the improved performance of materials have been increased, a variety of attempts have been made in order to develop the advanced material for high temperature applications. Aluminum alloys,[1] which bear new and unusual microstructure with improved engineering properties in place of titanium alloy, have currently been developed using the concepts of mechanical alloying to extend their use for high temperature applications up to 350°C.[2] Atomized and mechanically alloyed alloys can be formed to the uniform and thermally stable dispersoids in the aluminum matrix and result in significant microstructural variations. Mechanical alloying is a ball-milling process. Powder particles are repeatedly fractured and welded to produce homogeneous powders, which, are good for mechanical properties and prevent rapid decrease at elevated temperature. Unfortunately, although the rapidly solidified Al-Fe-Cr alloy system appeared to be promising,[3] only an atomized aluminum alloy is not satisfactory due to lacking homogeneous microstructures and thermal stability.[4] For following consolidation of these powders into the form of a billet, the material is hot-extruded; this processing can produce an extremely fine grain size and a high dislocation density. But, the materials are annealed at approximately seven tenths of melting temperature (0.7Tm) to produce an extremely deleterious and needle-like intermetallic compound ($Al_{13}Fe_4$) and elongated grain structure. To enhance strength and thermal stability of this alloy, ODS Al alloys produced by atomized and mechanically alloyed powders have excellent resistance to deformation at high temperature,[5-6] but limited ductility. Consequently, they cannot be shaped by metal working methods but the as-hot-extruded condition. Before the deleterious and needle-like intermetallic compounds and coarse grains are formed, they can be formed. Characterization of the atomized and mechanically alloyed Ai-9.45Fe-4.45Cr powders and extrudate is of interest and not wholly reported on herebefore. The microstructural changes and mechanical properties of atomized and mechanically alloyed powders and extrudates of ultrafine powder with an entirely featureless microstructure with milling time and elements variation are shown.

The present study on both atomized and mechanically alloyed powders and extrudates of Al-9.45Fe-4.45Cr alloy was undertaken to examine the microstructural changes and mechanical properties and to understand the effects of milling time. During the course of the work it became apparent that this ultra-fine grained alloy provides an opportunity to examine certain aspects of good strength and thermal stability in order to use the high performance aluminum alloy for the elevated temperature applications.

2. Experimental procedure

The Al-9.45Fe-4.45Cr powders were produced in a pilot atomizer at RASOM (Rapidly Solidified Materials Research Center), nitrogen gas being used as an atomizing-gas in order to increase cooling rate and prevent too thick and agglomerated oxidation of powders. The as-atomized powders (below 90 μm) were blended with 25 g of the organic surfactant ethylenebisdisteramide ($C_{38}H_{74}ON$) to prevent excessive agglomeration of the powder during ball milling. Each of batches was placed inside 160 mm-diameter and 160 mm-length hardened stainless steel vials with 10 mm-diameter steel balls (ball-to-powder ratio of 10 : 1) in air. Mechanical alloying was carried out at various times up to 240 hours. Based on the characterization studies during mechanical alloying of the rapidly solidified Al-8.4wt%Fe-3.4wt%Ce reported elsewhere,[11,12] the steady-state processing conditions were made after milled powders for 180 minutes.

During below MA, the destabilization of the crystalline phase is thought to occur by the accumulation of the point and lattice defects and antiphase boundaries. Characterization of the milled powders had been conducted by using XRD, SEM and Photoelectrons Spectroscopy (XPS) techniques. The analyzed values of chemical composition by XPS are shown in Table 1. The XRD studies had been done with a diffractometer using Cu Kα radiation at 30 kV and 50 mA. Comparing the peak positions and intensities those listed in the JCPDS files did identification of the phases. Microstructure has been examined by using both a PME-3 optical microscopy (OM) and a JEOL JSM 5410 scanning electron microscope (SEM) at 30 kV. Oxygen contents may affect the final materials. To determine the in-depth distribution of the elements, Depth profile of atomized and mechanically alloyed powders was conducted with an ESCALAB 210 XPS using Mg K radiation at 50 mV. Chemical compositions of the dispersed phases were determined with the aid of EDS attached to SEM at 30 kV.

Table 1. Chemical composition of rapidly solidified powders and ball-milled.

Fe	Cr		O	Al
Alloy(i)	9.45	4.45	0.227	bal.
Alloy(ii)	9.45	4.45	2.56	bal.

Transmission electron microscopy (TEM) investigations were done on thin foils prepared by iron milling in a 10pct perchrolic acid-20pct glycerol-70pct ethyl alchol solution at 0°C for 2 to 3 minutes. The thinned specimens were examined in a JEOL JEM-2010 TEM operated at 200 kV. The hardness of these powders was measured using a MicroVickers hardness tester with a load of 25 g. Compressive tests were performed from room temperature and up to 470°C using a MTS.

3. Results and discussion

Fig.1 displays the depth profile of the atomized and mechanically alloyed Al-9.45Fe-4.45Cr powders in atomic percentages conducted with a XPS. Accumulated oxidation of Al-Si-X, only an atomized and mechanically alloyed Al-Fe-Cr powders were calculated to be 0.227,[7] 0.21 and 2.56% in weight respectively, the initial oxygen contents of atomized and mechanically alloyed powders are more than those of only atomized powders.

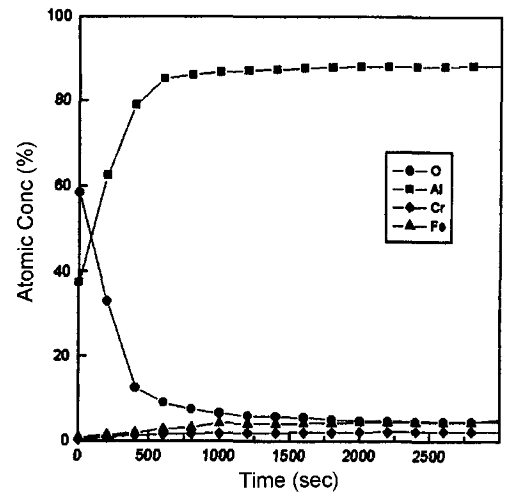


Fig. 1. Depth profile of powder showing the composition variation of Al, Fe, Cr and oxygen in the 500 thick layer of Al-9.45Fe-4.45Cr powders.

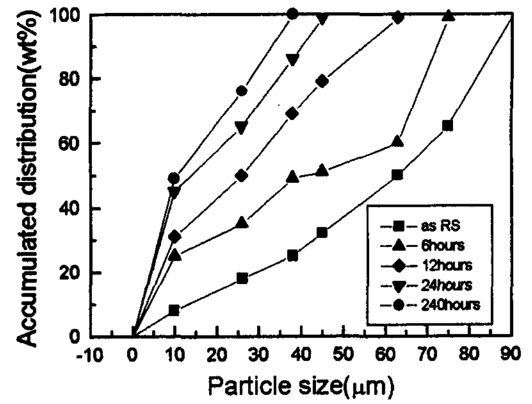
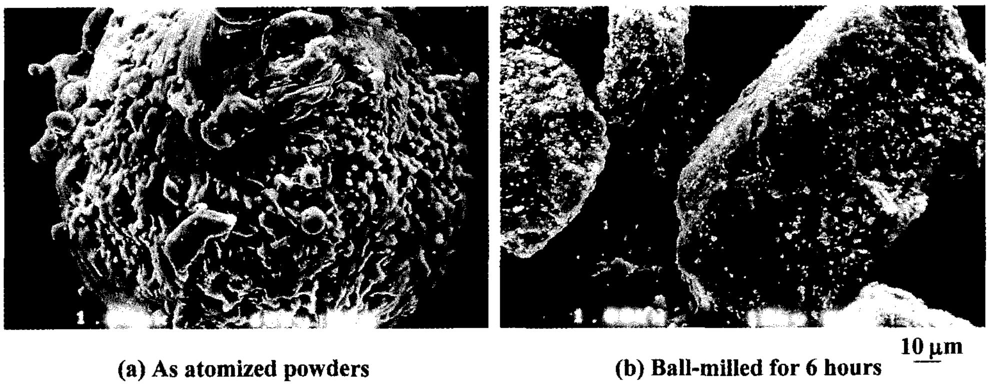


Fig. 2. Size distribution of the as-atomized Al-9.45Fe-4.45Cr powders and mechanically alloyed powders.

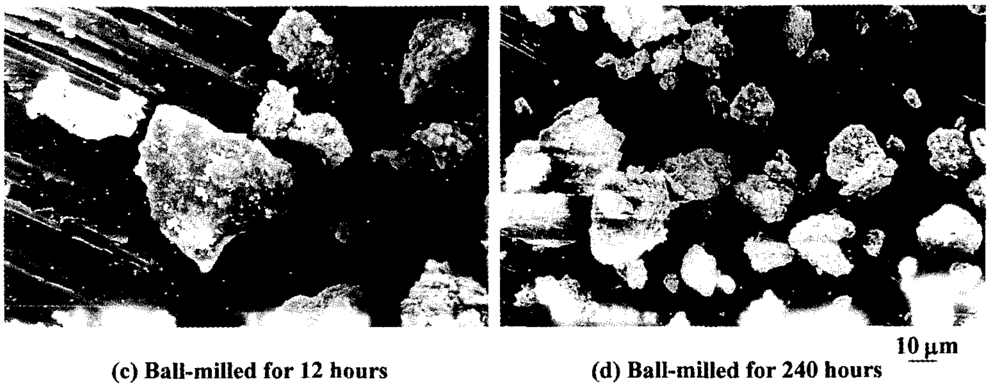
The elements in the atomized and mechanically alloyed Al-9.45Fe-4.45Cr powders have a great tendency to oxidation. During mechanical alloying, as powders particle size comes to finer, oxide of atomized powder is produced and fractured.

Fig. 2 and Fig. 3 show the size distribution of the powder investigated, which were obtained by



(a) As atomized powders

(b) Ball-milled for 6 hours



(c) Ball-milled for 12 hours

(d) Ball-milled for 240 hours

Fig. 3. SEM morphology of powders as atomized (a) and ball-milled for 6 hours (b), 12 hours (c) and 240 hours (d)

using non-linear least square analysis and SEM. The mechanical alloying depends on the milling parameter such as processing time and so on. According to Fig. 2, as the powders were mechanically alloyed for 6, 12, 24 and 240 hours, the medium diameters of powders were calculated 60, 42, 19 and 13 μm respectively and the size distribution was narrowed along with ball-milling time. It has already been known that the smaller the only atomized powder particle, the faster the cooling rate and the smaller the atomized and mechanically alloyed powder particle, the finer the microstructure and the more extensive the solid solubility due to creation of lattice defects (vacancies, dislocations and grain boundaries, etc) resulted from a deviator stress. During the milling operation, the present powder particles were repeatedly flattened, fractured and rewelded. Because fracture predominated, very fine particles formed, on the other hand, as partial welding took place in the powder particles which were mechanically alloyed for 240 hours, large powder particles partly produced. Excessive welding, most common during mechanical alloy of the ductile powders, could be avoided by using the organic surfactant ethylenebisdisteramide, by using the proper atmosphere or by lowering the mill temperature.

Phase identification in these alloys was performed by X-ray diffractometer. Binary alloys of Al with Fe, and Cr have a eutectic reaction and a peritectic reaction, crystallizing the Al solid solution and $Al_{13}Fe_4$ compounds at 1.8 wt%Fe and 655°C, and the Al solid solution and $Al_{13}Cr_2$ compounds at 3.2wt%Cr and 660°C under the equilibrium condition, respectively. The solid solubility of Fe and Cr in Al matrix is about 0.04 wt% at 655°C and about 0.77 wt% at 660°C, respectively. Cr binary alloys show significant solid solution strengthening and are resistant to the precipitation and coarsening of dispersoids at temperatures up to 400°C(627 K). The precipitation and coarsening of $Al_{13}Cr_4$ happened at 475°C.[6] Solid solution extensions are achieved in many alloy systems by far-from-equilibrium processing methods such as mechanical alloying. Although the alteration of the α-Al lattice parameter is complicated by the situation of iron atoms (smaller than aluminum atoms) and Cr atoms in the Al matrix, it is clear that the solubility of iron and Cr in Al matrix under the condition of mechanical alloying can possibly be enlarged to 4.5 at.% and 5.0 at.%, respectively. These solid solution extensions may be achieved with rapidly solidified and mechanically alloyed Al-Fe-Cr alloy (ternary alloy) as inter-diffusion process and reached a supersaturation and give rise to asymmetrical α-Al line broadening, shift indicating fine structure and Fe and Cr supersaturation, respectively. They are higher than those in aluminum under that of rapid solidification. These solid solution extensions may be achieved with milling time as inter-diffusion process and reached a supersaturation. This solid solubility level has been determined from the changes in the lattice parameter values calculated from the shift positions in the X-ray diffraction patterns. Fig. 4 shows X-ray diffraction patterns for the present powders after different milling time. In consequence of these patterns, the lattice parameter values calculated from the Al 1 diffraction after separation from the Al 2 diffraction exhibit 0.40504, 0.40503, 0.40501 and 0.40501nm with 6, 12, 24 and 240 hours, respectively. When the Al matrix is had addition to Fe or Cr elements, the

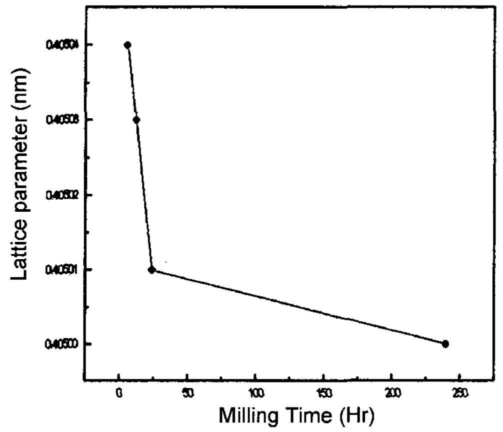
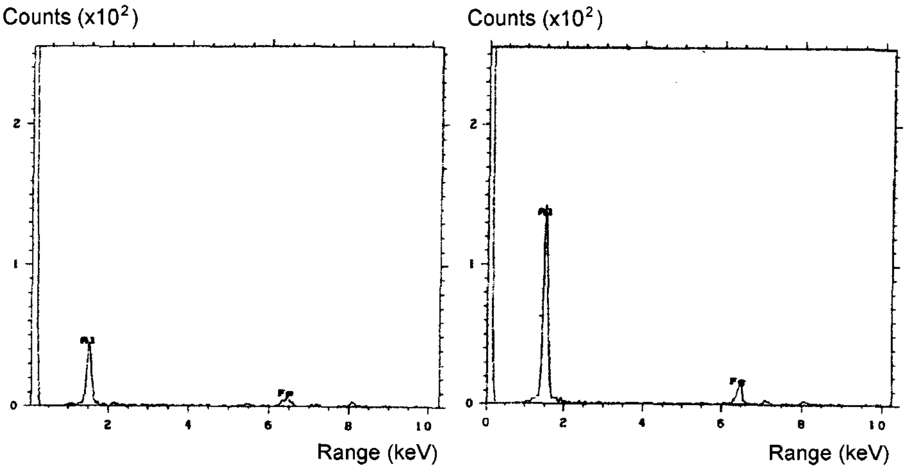


Fig. 4. Lattice parameter calculated by X-ray diffraction for according to milling times.

causes for the alteration of the lattice parameters in the atomized and mechanically alloyed Al alloys are very difficult and complicated. But the decreased lattice parameters of this Al matrix was mainly explained by a drainage effect due to intermetallic[8)] and the increased supersaturation level due to mechanical alloy. Both disordered and ordered intermetallics have been synthesized by and mechanical alloy. In some case, the intermetallics were synthesized directly by mechanical alloying without thermal heat treatment. For example, intermetallic compound has been found to form directly on and mechanical alloy in Al-rich Al-transition metal systems($A_{15}Fe_2$, $A_{13}Fe$ etc). The formation of ordered intermetallics may be assumed that a phase will exist either in the ordered and the disordered condition depending on the balance between atomic disordering introduced

(a) Atomized extrudate

(b) Ball-milled extrudate



(c) EDX of $Al_{13}Fe_4$

(d) EDX of Al_6Fe

Fig. 5. Transmission electron micrographs of atomized extrudate (a), ball-milled extrudate (b), EDX of $Al_{13}Fe_4$ (c) and Al_6Fe (d)

by mechanical alloy.

To get a better understanding of the morphology and chemistry of the different phases present, TEM investigations were performed. Some typical electron micrographs from not only as atomized extrudates but also ball-milled extrudates are displayed in Fig. 5.

The presence of the dispersoids that are coarse and needle-like suggests that the undercooking level be so low that high supersaturation was not possible. But the fine and optically featureless dispersoids in the ball milled bulk does that high supersaturation be possible.

Fig. 6 shows the average microhardness values measured on the powder particle sections. In this test, hardness of ball-milled powders varies with ball-milling time. Generally, It should be noted that the section area does not necessarily present the powder particle size, but the microstructural transition with milling time gives significant variation in hardness. The hardness of the as-atomized powder particles smaller than 10 μm is apparently good, but such a small size range is not presently feasible. To produce this small particle size, it is necessary that powders be mechanically alloyed by the ball milling. However, the specific area and oxygen contents of the powder would be greatly increased and the embrittlement resulting from the presence of oxide in the consolidated materials[9] would be an obstacle to the commercial products of this alloy. But controlled and dispersed ultrafine oxides in the ball-milled powder particles may enhance the yield strength and the ultimate compressive strength. Hardness of this powders can be explained by the mechanism including fine cell hardening, fine and featureless dispersoid hardening, solute hardening and dispersed ultrafine oxide hardening, which operate in the ball-milled and homogeneous powders. Thus, the uniform deformation of the homogeneous powders, which are different from the as-atomized and inhomogeneous powders during consolidation, can be expected.

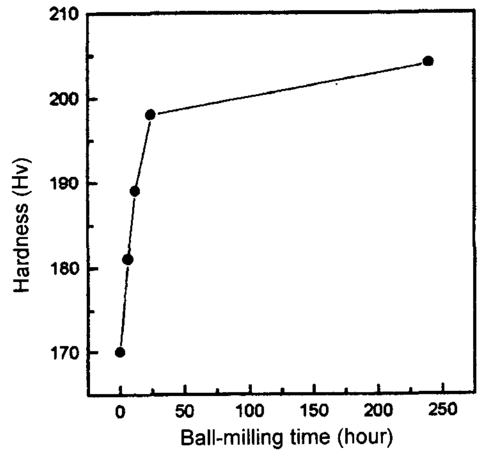


Fig. 6. Variation of microhardness in the powder particles section with ball-milling time.

Monotonic tensile and compressive tests results are given in Table 2 and Fig. 7, respectively. These room and high temperature strength and ductility values are comparable to those published[10,11] for Al alloys strengthened by dispersions of

Table 2. Results of tensile tests performed on the extruded powder tested at 20, 200, 343 and 420°C

Alloy (Rapidly Solidified)	Test Temperature °C(K)	Yield Strength (0.2% Offset)	Ultimate Tensile Strength(MPa)	Elongation (%)
Al-9.45Fe-4.45Cr	RT	451	518	1.649
	200(473)	378	427	1.353
	343(616)	303	336	1.226
	420(693)	197	214	1.472
Al-7.1Fe-6.0Ce(12)	RT	462	493	12.1
	149(422)	389	425	6.1
	232(505)	317	353	6.7
	316(585	215	248	6.6
Al-9.0Fe-2Cr-0.7Mn(12)	RT	520	586	1
	400(673)	249	324	6.8

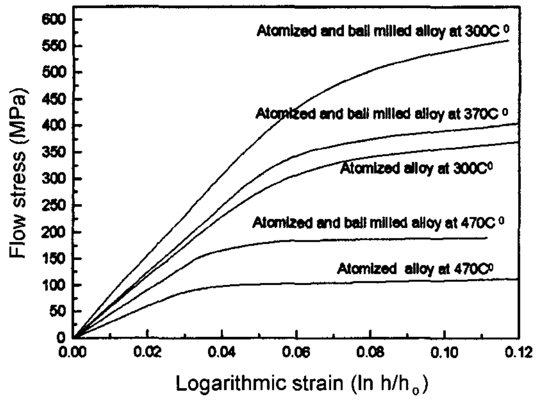


Fig. 7. Compressive test of atomized and ball-milled Al-9.45Fe-4.45Cr alloy and atomized Al-9.45Fe-4.45Cr alloy.

intermetallic particles which decrease second phase particle coarsening. Atomized aluminum alloy, which bears a small volume fraction, large space and needle shape of dispersoids commonly, consists of large diameter powders. The correlation between strength and inter-particle spacing exhibited that inter-particle spacing in the finer and more dispersoids control the strength of these alloys. Therefore, because the particle spacing at room temperature is smaller than it at elevated temperature, the former of strengths is better than the latter of them, even if both lower fabrication temperature and higher solute content enhance strength by the decreasing inter-particle spacing.$^{11)}$ Atomized and ball-milled Al-9.45Fe-4.45Cr alloy doesn't rapidly decrease at temperature below 300°C(573 K). On the contrary, they decreased with rising temperature higher than 300°C(573 K). The coarsening of dispersiods resulted in the decreased tensile strength and hardness. But atomized and ball-milled Al-9.45Fe-4.45Cr alloy with higher volume fraction of thermally stable intermetallics had much better mechanical properties than only atomized Al-9.45Fe-4.45Cr alloy. In this regard, microstructure is close relation to mechanical properties. In the present alloys, the control of ductility is also very difficult due to difficult structure and a lot of dispersiods.

Analysis of data in Table 2 exhibits that two factors control ductility in these alloys, that is, one is that how well the powders particulates are bonded and how the fine oxides are distributed, the other is the size distribution and volume fraction of second phase particles. Large particles easily nucleate voids and allow more growth before coalescence, while small particles difficultly nucleate voids and allow more rapid coalescence.$^{12)}$ The extrudates of Al-9.45Fe-4.45Cr demonstrate special low ductility in this extrusion condition due to high volume fraction of intermediate compounds and dispersoids, which result from the increased chromium contents and ball milling. The influence on ductility resulting from inter-particle oxides can be minimized by the use of sufficient extrusion ratio (25 : 1 in these alloys). Ductility is controlled by the non-sharable second phase in these present alloys. It is noted that the increasing volume fraction of second phase finally results in drop of it.

4. Conclusion

Machanical alloying is a simple and versatile techinque capable of producing different types of metastable effects in variety of alloy system. Based on the results shown above, the following conclusions have been drawn.

1) Atomized and ball-milled Al-9.45Fe-4.45Cr alloy can provide a high volume fraction of second phase.

2) Atomized and ball-milled Al-9.45Fe-4.45Cr alloy with high volume percentage of second phase particle results in mechanical properties, i.e. ductility decreased and strength increased with volume percentage of second phase particles.

3) As the temperature increased, atomized and ball-milled Al-9.45Fe-4.45Cr alloy with higher volume fraction of thermally stable intermetallics and dispersoids had much better mechanical properties than only atomized Al-9.45Fe-4.45Cr alloy.

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