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SURFACE CARBON FILMS ON Al-Cu-Fe**QUASICRYSTALLINE POWDERS**

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In order to study the unique properties of quasicrystals, it is necessary to form dense, homogenous monoliths of these alloys. Unfortunately, Al-Cu-Fe quasicrystalline alloy ingots prepared by conventional casting techniques result in large scale chemical inhomogeneities^{1,2} which contain numerous cracks due to differential thermal contraction between the various phases during cooling. Thus a powder metallurgical approach using gas atomized (GA) powders is being pursued in order to form large samples of phase pure Al-Cu-Fe quasicrystal. GA samples of specific compositions and sizes are hot isostatic pressed (HIPed) to form dense monoliths. The effects of surface contamination of GA powders, which may inhibit particle-to-particle sintering and may also increase second phase contamination in the HIPed piece, is being studied by scanning Auger microprobe (SAM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

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It has been demonstrated that gas atomization (GA) and powder processing methods can be used to form multigrain nearly single phase monoliths of Al-Cu-Fe quasicrystal.³ GA is a rapid-solidification process used here to obtain chemically homogeneous spherical powders (~5 to 100 μm).⁴ Hot isostatic pressing (HIPing) promotes the formation of particle-to-particle bonds and sintering leading to enhanced densification. Incorporation of surface contamination from the powder coatings to the HIPed monolith in the form of inclusions may have an adverse affect on the materials properties. Therefore, characterizing and controlling the coating on the powder is an important step. The coating composition and thickness of GA powders can be controlled by the choice of atomizing gas as demonstrated with pure Al.⁵

The pure Al sample was melted in a hard-fired alumina crucible under an Ar atmosphere, the atomization chamber was then evacuated, back-filled and atomized with N₂ (GARS-3). GA samples of Al-Cu-Fe were produced from a chill cast ingot which was ground and used as a feed stock with Ar gas as the atomizer. Bulk analysis was performed on each sample by inductively coupled plasma atomic emission spectroscopy (ICP) (Table 1). SEM and Auger electron spectroscopy (AES) were performed on each sample using a Physical Electronics SAM 660 to determine the coating composition and thickness. AES analysis consisted of spot mode for beginning composition, followed by depth profile at a rate of 12 nm/min to determine the thickness of the coating layer.

The Al sample was cold isostatic pressed (CIP) and sintered for 1 hr at 550 °C in vacuum. The quasicrystal powders were HIPed at various pressures and temperatures in evacuated and sealed Cu or stainless steel cans. Samples (~2 x 2 x

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Table 1. ICP analysis of GA samples used in this study.

at %	Al	Cu	Fe
GA-165-P	64.96	23.05	11.99
GA-2-1-35	63.83	24.44	11.73
GA-2-282	65.0	23.0	12.0
GARS-3	99.9	<0.056	<0.032

performed. Some samples were examined by TEM to evaluate the prior particle boundaries and mode of deformation.

2. Results and Discussion

Pure Al is used as a model system to show the effect of GA processing conditions on the surface coating of the powders. The surface oxide film can be controlled by choice of the atomization gas and the pour temperature. The measured oxide boundary on CIP samples compares well between direct TEM measurements and AES depth profiling, validating depth profiling method for this system (Fig. 1).

In the more complex Al-Cu-Fe system, C and O are both found on the surfaces of the atomized powders (Fig. 2). The C is in part due to atomizing conditions and atmospheric contamination. In the first run (HIP1), ~75 μm particles (GA-2-282) were HIPed at 750°C with 70 MPa confining pressure. This material had variable thickness of C and O surface films, and in some areas greater than 150 nm thick

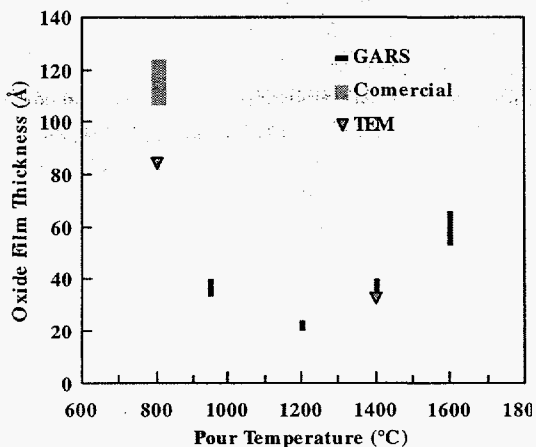


Figure 1. Oxide film thickness as a function of pouring temperature for gas atomized Al using N₂ (GARS) and for commercially obtained powder atomized in air.

The powders show no more than 45% at.% C which is up to a

10 mm) were fractured in situ in the Auger chamber at 3×10^{-10} torr. Auger spectra, maps and in some cases depth profiling was

(Fig. 3) resulting in C films persisting on the clearly obvious prior-particle boundaries in the *in-situ* fractured HIPed samples (Fig. 4). Analysis of the HIPed samples by energy dispersive spectroscopy shows that the C is only at the fracture surface in measurable concentrations. SAM analysis of the fracture surfaces gives similar results where C on fresh fracture surfaces reach 70 at.% at the prior particle boundaries verses 14% at.% in particle centers. However, the C in the particle centers is due to C migration across the sample surface over time. The powders show no more than 45% at.% C which is up to a

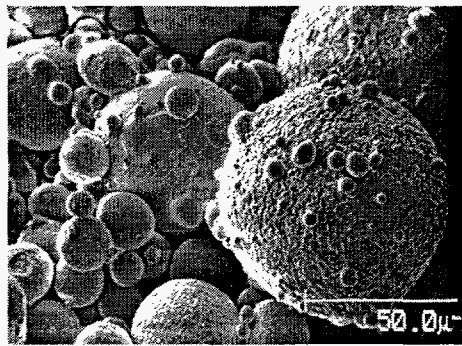


Figure 2. SEM micrograph of the Al-Cu-Fe GA powders (GA-2-282) used in HIP run 1.

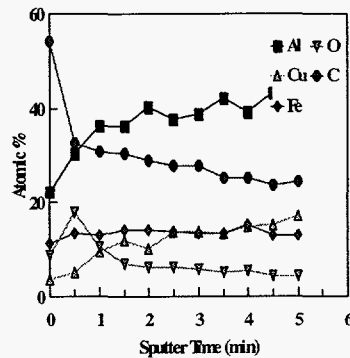


Figure 3. AES depth profiling of one of the Al-Cu-Fe GA particles (GA-2-282) in Fig. 1.

depth of 150 nm. The integrity of HIP samples from these particles was not good, as indicated by the numerous cracks throughout the sample.

In the subsequent HIP runs (HIP2 and HIP3), smaller particles were used (~ 10 μm) (GA-165-P and GA-2-1-35) (Fig. 5) as well as a higher confining pressure (315 MPa) and temperature (850°C). The starting composition for HIP3 was slightly lower in Al and higher in Cu than HIP2 (Table 1). However, both samples show considerably thinner C and O surface films on the GA powders (Fig. 6) compared to powders used in HIP1. The *in-situ* fracture surfaces of these samples show lack any distinguishing characteristics of the prior particle boundaries, indicating sintering or diffusive bonding is complete.

3. Conclusion

Proper control of the powder processing can give rise to homogeneous, dense monoliths in the Al-Cu-Fe quasicrystal system. These results clearly indicate that cleaner surfaces of the starting powders results in less contamination of the HIPed

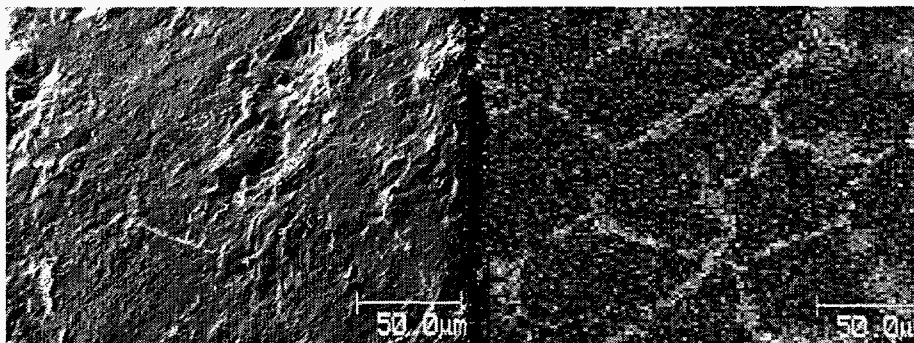


Figure 4. a) SEM micrograph of the *in-situ* fracture surface of HIP1 and corresponding b) AES map showing a persistent C coating on the prior particle boundaries.

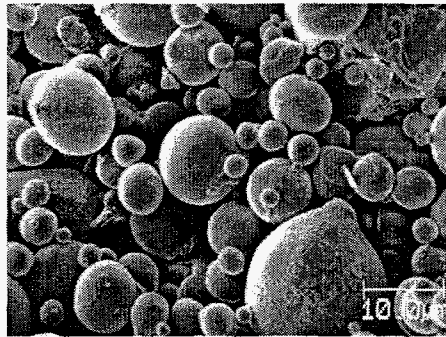


Figure 5. SEM micrograph of the Al-Cu-Fe GA powders (GA-2-1-35) used in HIP run 3.

sample. The higher processing temperature and pressure in conjunction with cleaner particle surfaces eliminated indications of the prior particle-to-particle boundaries. Although these samples are not phase pure after HIPing, they can be converted to homogeneous monoliths with subsequent heat treatments.⁶

Acknowledgments

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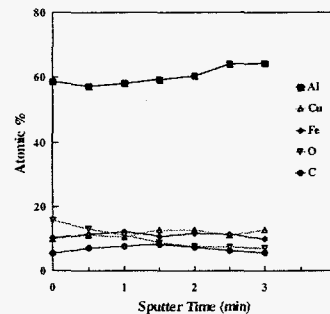


Figure 6. AES depth profiling of the Al-Cu-Fe GA particles used in HIP3.