ISSN : 0974 - 7486 Volume 14 Issue 7

Materials Science
An Indian Journal
Full Paper

MSAIJ, 14(7), 2016 [235-239]

Microstructure properties of rapidly solidified Al-5Cr alloy

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ABSTRACT

Rapidly solidified Al-5 wt.% Cr alloy has been prepared by the melt spun technique in form of ribbons. Microstructure and subsequent structural changes were examined as a function of heat treatment using XRD technique and Rietveld analysis. Metastable $Al_{0.983}Cr_{0.017}$ and α-Al phases have been observed with 98.1 and 1.9 wt.% contents in the as melt spunribbon, respectively. During heat treatment process at 250 °C for 20h, content of $Al_{0.983}Cr_{0.017}$metastable phase decreased to 56 wt.% while content of α-Al increased to 39.5 wt.%. In addition, $Al_{45}Cr_{7}$ phase has beenobserved in the heat treated ribbon with content of 4.5 wt.%. Lattice constant of the as melt spun $Al_{0.983}Cr_{0.017}$metastable phase has been observed to be less than the equilibrium value referring to that part of Cr has been dissolved in the $Al_{0.983}Cr_{0.017}$metastable phase. After heat treatment, this value increased to be close to the equilibrium one owing to Cr precipitations. Rapid solidification had a great role for forming the $Al_{0.983}Cr_{0.017}$ metastable phase.

KEYWORDS

Rapid solidification;
Al base alloys;
Metastable phases;
XRD;
Redfield analysis.

INTRODUCTION

Rapid solidification is a processing technique in which unique properties and microstructures can be obtained which are not found through conventional processing. Rapid solidification involves cooling of metallic melts at rates >10^4 K/s and results in significant microstructural and constitutional changes. The microstructural modifications include grain refinement and reduced segregation effects while the constitutional changes include formation of supersaturated solid solutions, and metastable crystalline intermediate and amorphous phases. These effects, either alone or in combination, have improved the mechanical behavior and performance of the rapidly solidified alloys and these results were especially significant for lightweight metals and have been well documented in the literature[1–5]. Chromium is known to form large size intermetallic particles with aluminum, which render the alloy quench sensitive, and the alloy system was also associated with excellent corrosion and oxidation resistance[6-10]. Rapidly solidified Al-Cr alloy is known to form metastable supersaturated solid solution in the range of lower chromium content and icosahedral quasicrystals over a wide composition range from about 5 to 16 at% Cr. In addition, Al-Cr alloy is one of the most familiar materials and has been well investigated[11-21]. The purpose of this work was to investigate the microstructure characteristics of rapidly solidified Al-5 wt. % Cr ribbon, prepared by the single roller method.

EXPERIMENTS

Material preparation

Al-5 wt. % Cr alloy was prepared from 99.8 w% pure Al, 99.75 wt.% pure Cr. The ingots were melting in a muffle furnace and poured into a graphite mold after the homogenization process to produce rods of 25 mm in length and 4 mm in diameter. Long uniform ribbons of thickness 50 μm and width 2 mm were prepared by melt spinning. A stream of the molten alloy, at 1100 °C, was ejected by argon gas at a gauge pressure of 1.5 bars from a silica tube with a 0.4 mm orifice diameter. The melt jet fell on a copper disc of 18 cm diameter coated by chromium, rotating at 2950 rpm. The estimated cooling rate was about 10^5 K/s. The produced ribbons were fairly uniform. Deviations of 0.05% mm and 3 μm in width and thickness were observed from the whole length of ribbons.

Material characterization

XRD patterns were performed using a 1390 Philips diffractometer with filtered Cu Kα radiation λ=1.541A0. The X-ray samples were performed from a short length stuck on a glass slide using Vaseline. Rietveld X-ray diffraction analysis[22] was carried out by the X'pert HighScor 2004 program and the Pseudo-Voigt peak shape function. The reliability of the refinement results was judge by the pattern R factor (R_p), the weighted pattern R factor (R_{wp}) and the 'goodness of fit' (GOF = $(R_{wp}/R_{xp})^2$)[23]. Initial structure parameters of all phases used for Rietveld method in this study were from ICCD (Inorganic Crystal Structure Database) cards. The parameters that had been refined simultaneously include: scale factors, zero point shift, lattice parameters, atomic coordinates, atomic sites occupancies, isotropic or anisotropic temperature factors, profile shape parameter, FWHM (Full Width at Half Maximum) parameters, asymmetry and preferred orientation parameters. The total parameters to be refined of Al-5Cr melt spun and annealed ribbons were 11 and 56 respectively. Rietveld method is becoming progressively more popular for microstructure characterization of materials. It is common practice to estimate domain size and strain values from the refined profile width parameters. Moreover, weight fractions of all phases in multiphase sample can be calculated directly by their scale factors which can be obtained by Rietveld fitting. The relationship between the fraction (W_i) for each phase i and its scale factor (S_i) determined is obtained from the following relation[23]:

$$W_i = \frac{S_i(ZMV)}{\sum_{i=1}^{n} S_i(ZMV)_i}$$

where Z, M, V are the numbers of formula unite cell, unit molecular weight of the formula and unit cell volume of phase i in a mixture of n phases respectively. Weight fraction of all phases observed in this work, crystallite size of and micro-strain% of α-Al have been estimated using Rietveld X-ray diffraction analysis.

RESULTS AND DISCUSSION

Figure 1 shows XRD patterns of Al-5Cr melt spun and annealed ribbons. For the melt spun ribbon, it is obvious that it consists of a single phase. It was proved that this single phase is $Al_{0.983}Cr_{0.017}$. In addition, there are no intermetallic compound has been formed between Al and Cr elements. After annealing at 250 °C for 20 h, the XRD lines are shifted to lower diffraction angle as shown in the inside figure where the highest peak in the XRD pattern was splitted into two overlapped peaks. Moreover, there was an addition phase has been observed which defined as $Al_{45}Cr_7$ phase. The Rietveld method considers overlapped peaks and the effect of preferred orientation therefore, this method could be powerful technique to quantitative phase analysis of nanocrystalline melt spun alloys. Moreover, overlapped XRD peaks corresponding to different phases can be separated and quantity of the different phases

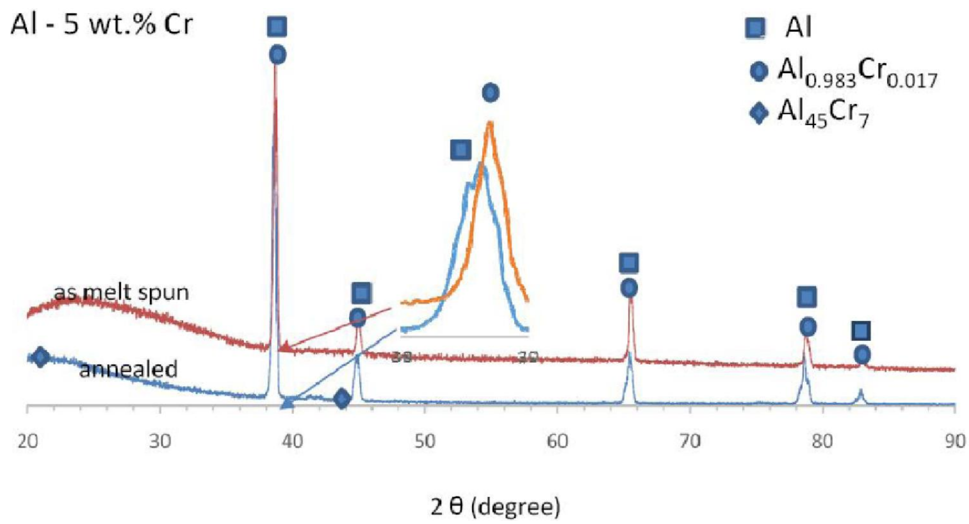

Figure 1 : XRD patterns of as melt spun and annealed (250°C/20h) Al-5 wt.% Cr ribbons

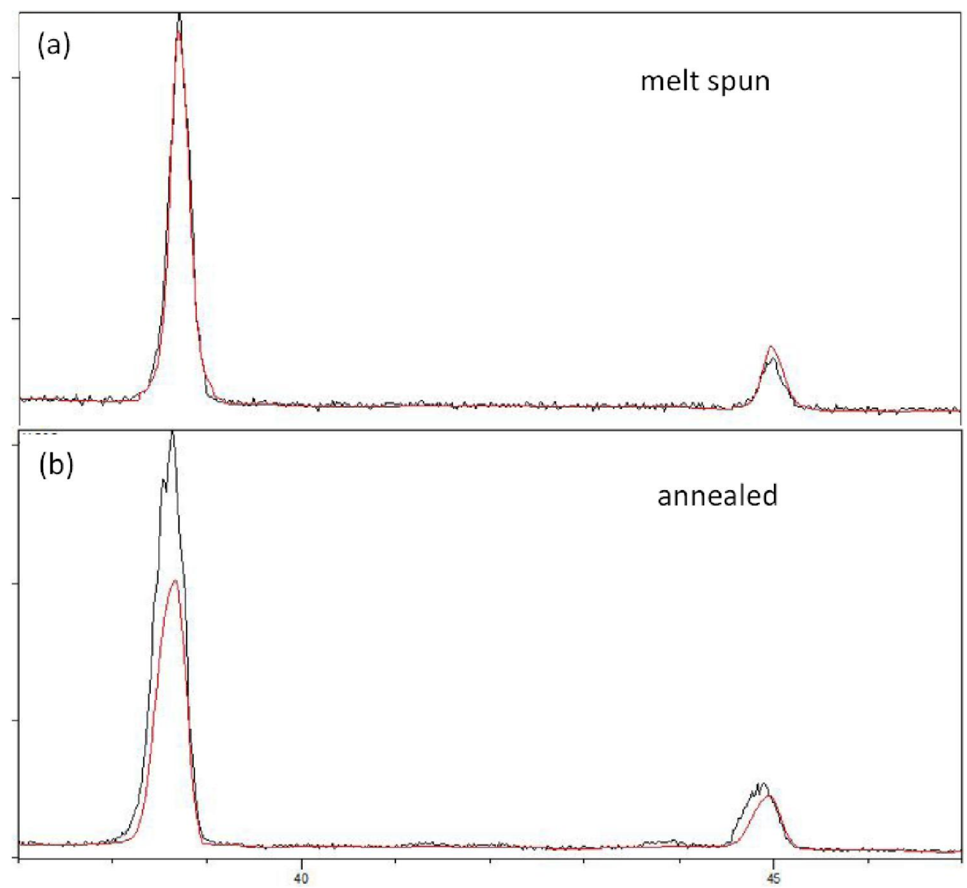

Figure 2 : Comparison between the observed XRD pattern (black) and the calculated XRD of $Al_{0.983}Cr_{0.017}$(red) forthe as melt spun (a) and annealed (b) ribbons

can be determined.

Figure 2 and 3 show comparison between observed and calculated XRD pattern obtained from the Rietveld analysis for $Al_{0.983}Cr_{0.017}$ and Al phases separately. It is obvious that, content of the $Al_{0.983}Cr_{0.017}$ phase in melt spun state is greater than that in the annealed ribbon, while content of the Al phase in melt spun ribbon is less than that in the annealed ribbon. This means that, the rapid solidification had a great role on formation of the $Al_{0.983}Cr_{0.017}$ phase.

During the annealing process Cr atoms precipi-

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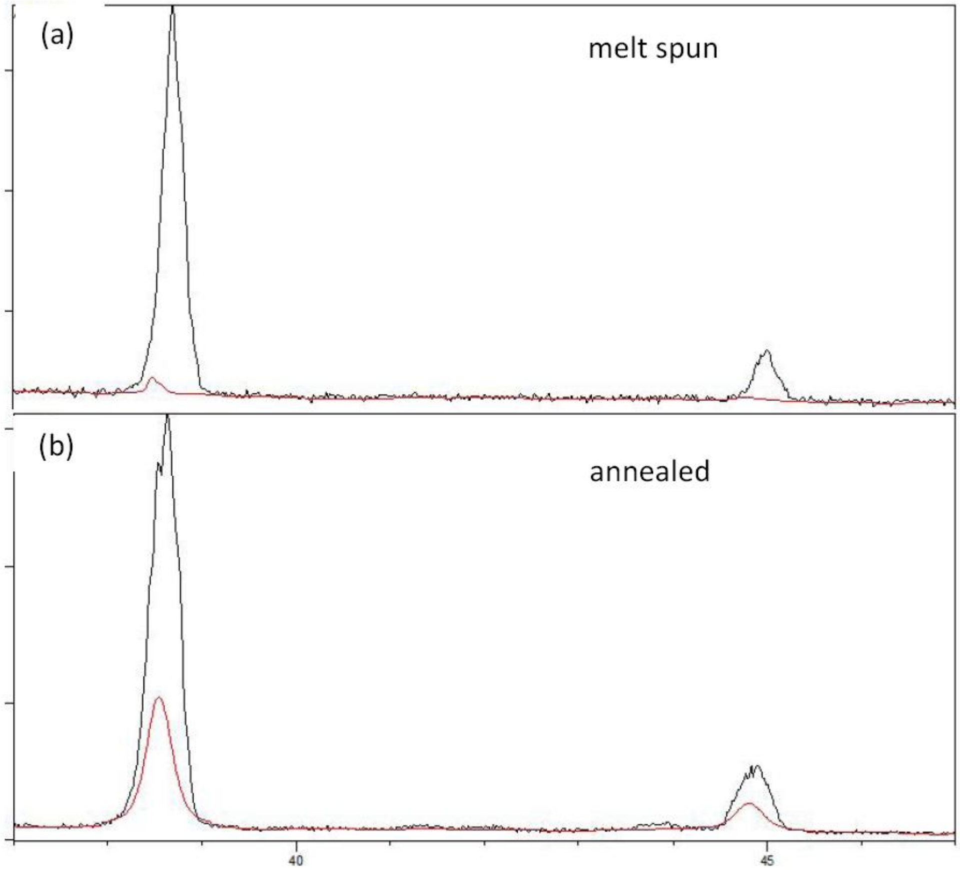

Figure 3 : Comparison between the observed XRD pattern (black) and the calculated XRD of α-Al(red) for the as melt spun (a) and annealed (b) ribbons

TABLE 1 : Contents in wt. % of $Al_{0.983}Cr_{0.017}$, Al and $Al_{45}Cr_7$ in the as melt spun and annealed Al-5Cr ribbons

	$Al_{0.983}Cr_{0.017}$ wt.%	Al wt.%	$Al_{45}Cr_7$ wt.%
As-melt spun	98.1	1.9	-
Annealed	56.0	39.5	4.5

TABLE 2 : Redfield analysis results of $Al_{0.983}Cr_{0.017}$ and Al in as melt spun and annealed states

Phase	As-melt spun Lattice constant crystal size ε% (nm) (nm)	Annealed Lattice constant crystal size ε% (nm) (nm)
$Al_{0.983}Cr_{0.017}$	0.4026500 42.1 0.045	0.4031788 55.2 0.038
Al	0.4045831 - -	0.4046494 45.5 0.116

tate from the metastable $Al_{0.983}Cr_{0.017}$ phase and therefore the content of $Al_{0.983}Cr_{0.017}$ phase decreases and the Al phase increase. TABLE 1 shows the contents of the different phases observed in the melt spun and annealed ribbons as deduced by the Rietveld analysis. In melt spun ribbons, the wt.% of $Al_{0.983}Cr_{0.017}$ phase and Al are 98.1% and 1.9% respectively, while their wt.% are 56 and 39.5 in the annealed ribbons, respectively. The precipitated Cr atoms appeared in form of an intermetallic $Al_{45}Cr_7$ phase. TABLE 2 shows the lattice constant, crystal size and lattice strain of $Al_{0.983}Cr_{0.017}$ and Al in melt spun and annealed ribbons. It is known that the lattice constant of casting $Al_{0.983}Cr_{0.017}$ and α-Al phase are 0.40365 and 0.40494 nm (equilibrium values) respectively. However, the lattice constant of melt spun $Al_{0.983}Cr_{0.017}$ and α-Al phases are 0.4026500 and 0.4045831 nm as shown in TABLE 2. It is obvious that the lattice constant of both phases have been decreased. This decrease of lattice constant of both phases can be related to the solid solution of Cr in the matrix of both phases. However, the lattice constant of both phases increase to approach the equilibrium values in annealed ribbons. The crystal size and lattice strain % of melt spun $Al_{0.983}Cr_{0.017}$ phase are 42.1 nm and 0.045 respectively, while for melt spun Al phase they could not be determined because of the little content of Al. During the annealing process, the lattice strain % of $Al_{0.983}Cr_{0.017}$ phase decrease from 0.045 to 0.038. Regarding the Al phase, the lattice strain records value as high as 0.116. It is worth to mention that the annealing temperature (250 °C) was not high enough to dissolve the Cr atoms completely, since the Cr atoms dissolve at temperature around 500 °C. One can mention that, rapid solidification had a great role for forming the $Al_{0.983}Cr_{0.017}$ metastable phase.

CONCLUSION

Rapidly solidified Al-5Cr has been successfully prepared using melt spun technique in form of ribbons. The obtained as melt spun and annealed ribbons have been described using XRD technique and analyzed by Redfield analysis. Microstructure of as melt spun ribbons has been proved to be a single metastable phase of $Al_{0.983}Cr_{0.017}$ (98.1 wt.%)with a little amount of α-Al (1.9 wt.%) and no intermetallic compounds have been observed. As a result of the thermal treatment at 250 °C for 20h, Cr atoms precipitate from metastable phase of $Al_{0.983}Cr_{0.017}$forming a new phase of $Al_{45}Cr_7$ with 4.5 wt.% content. Moreover, the contents (wt.%) of $Al_{0.983}Cr_{0.017}$ decreased to 56.0 while α-Al increased to 39.5. The crystal size and lattice strain % of melt spun $Al_{0.983}Cr_{0.017}$ phase were 42.1 nm and 0.045. Rapid solidification had a great role for forming the $Al_{0.983}Cr_{0.017}$ metastable phase.

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