MICROSTRUCTURAL EVOLUTION OF MELT-SPUN Al-(Fe,Nb)-Si RIBBONS

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ABSTRACT: In the present work Al-(Fe-Nb)-Si ribbons alloys with different Fe/Nb ratio were made by melt spinning. The microstructural evolution and the microstructures were investigated by means of differential scanning calorimetry and x-ray diffraction. Nb addition is very effective to stabilize the bcc $\alpha$-$\text{Al}_{13}(\text{Fe,Nb})_3\text{Si}$ silicide. However, its lattice parameters is smaller than that of the $\alpha$-$\text{Al}_{13}(\text{Fe,V})_3\text{Si}$ silicide in the AlFeVSi systems. Unlikely AlFeVSi systems in that alloy systems it was observed a phase transformation in a temperature range of 400-490°C which was attributed to the extra $\alpha$-$\text{Al}_{13}(\text{Fe,Nb})_3\text{Si}$ silicide.

INTRODUCTION

Nanocrystalline materials produced by rapid solidification processing (RSP) have attracted considerable attention during the past few years. The generation of these new solids with ultrafine grains and large volume fractions of interfaces has resulted in their novel properties which are superior to those of conventional polycrystals. One such RSP alloy, Al-8.5Fe-1V-2Si prepared by Allied Signal exhibits promising mechanical properties at temperatures up to 350°C, attributed to the slow coarsening rate of the silicide $\text{Al}_{13}(\text{Fe,V})_3\text{Si}$ phase [1-4]. The slow coarsening rate is owing to the low solubility and diffusivity of transition metals in aluminum and also to the small lattice parameter mismatch between the aluminum matrix and the silicide phase. The silicide structure is a b.c.c packed two shell icosahedral cluster, where Fe and V occupies the same site in the cell. The same type of silicide can be stabilized by replacing the Fe by others transition elements such as Mn, Nb, Cr, Mo, Co [5-8]. Therefore, silicide similar to those of the Al-Fe-V-Si alloys are expected. However, the thermal stability of the alloys can be changed as a result of the lattice parameter misfit as well as others microstructural.

In the present work was explored the possibilities of obtaining a similar structure by substituting Fe by Nb elements. The effect of the Fe/Nb ratio on the microstructure was studied. The microstructural evolution and the microstructures were investigated by means of differential scanning calorimetry and x-ray diffraction.

EXPERIMENTAL PROCEDURE

The melt stock used consisted of 99.99% purity aluminum and iron, 99.9% purity niobium and 99.99% purity silicon induction melted in a water cooled cooper crucible under argon atmosphere. Al-8Fe-4Nb-1.5Si and Al-8Fe-1.6Nb-Si alloys ribbons was prepared by the single roller spinning process in a argon atmosphere. The circumferential velocity of the melt-spinning roll was 30m/s. An amount of 5-8g of section of master alloys were then melt spun, using a quartz crucible with the melting temperature at ~ 1050°C. The ejection pressure was 10mbar. The specimens was 2 mm wide and ~ 40µm thick. The microstructural evolution of the ribbons was studied by differential scanning calorimetry (DSC) employing Ozawa and Kissinger methods [9-11]. In this study the as-quenched and heat treated microstructure was investigated. The heat treatment was performed at 500°C for 70h using a tubular furnace under argon atmosphere. Identification of phases in the melt spun and heat treated ribbons was done by X-ray Cu-Kα.
RESULTS AND DISCUSSION

The DSC thermograms, as shown in Fig. 1 (a) and (b), were obtained at heating rates of 5, 10, 20 and 30°C/min over the range 30-500°C. The principal feature of DSC thermograms of melt spun Al-8Fe-4Nb-1.5Si and Al-8Fe-1.6Nb-Si ribbons was one exothermic reaction. This exothermic reaction is not observed in as-quenched Al-Fe-V-Si alloys [12].

![Fig.1 DSC thermograms for various heating rates showing the exothermic reaction in melt spun ribbons. (a) Al-8Fe-4Nb-1.5Si; (b) Al-8Fe-1.6Nb-1.5Si.](image)

The average peak temperature and heat effects associated with this transformation as a function of heating rates for both alloys are summarized in Table 1.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Heat (J/g)</th>
<th>Peak temp.(°C)</th>
<th>Heat (J/g)</th>
<th>peak temp.(°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-8Fe-4Nb-1.5Si</td>
<td>5.1</td>
<td>429.2</td>
<td>5.38</td>
<td>430.5</td>
</tr>
<tr>
<td>Al-8Fe-1.6Nb-1.5Si</td>
<td>4.3</td>
<td>443.5</td>
<td>5.025</td>
<td>445.4</td>
</tr>
<tr>
<td></td>
<td>3.1</td>
<td>457.1</td>
<td>4.25</td>
<td>459.6</td>
</tr>
<tr>
<td></td>
<td>-----</td>
<td>-----</td>
<td>4.02</td>
<td>463.4</td>
</tr>
<tr>
<td></td>
<td>2.9</td>
<td>465.5</td>
<td>-----</td>
<td>-----</td>
</tr>
</tbody>
</table>

The values of the activation energy of the microstructural changes employing Kinssinger-like method was determined by plotting the \(\ln(T^2/\phi)\) versus 1/T, as shown in Fig. 2.

![Fig.2 Plots of \(\ln(T^2/\phi)\) versus the reciprocal temperature of the observed microstructural changes in melt spun Al-8Fe-4Nb-1.5Si and Al-8Fe-1.6Nb-Si ribbons.](image)
The values of activation energy calculated by Kissinger and Ozawa method are listed in the table 2. In both alloys the activation energy value is, approximately, equal indicating the presence of the same phase transformation in the alloys. It is interesting to observe that the calculated activation energy, $Q$, is in good agreement with the reported activation energy for volume diffusion of Fe in aluminum, (190.1kJ/mol) indicating that the observed microstructural changes is controlled by volume diffusion rather than interface diffusion [13]. To determine the products of transformations, a detailed XRD analysis of melt spun and heat treated ribbons was performed, as shown in Fig.3.

Table 2 Activation energy of the phase transformations calculated by Kissinger and Ozawa methods.

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Activation energy (kJ/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Kissinger</td>
</tr>
<tr>
<td>Al-8Fe-4Nb1.5Si</td>
<td>204.4</td>
</tr>
<tr>
<td>Al-8Fe1.6Nb1.5Si</td>
<td>198.6</td>
</tr>
</tbody>
</table>

Fig.3 XRD spectra of the melt spun and heat treated Al-8Fe-4Nb1.5Si alloys ribbons.

From XRD spectra of the as-quenched melt spun ribbons was identified the diffraction peaks of bcc $\alpha$-Al$_{13}$(Fe,Nb)$_3$Si phase, with lattice parameters of 1.256nm, which is slightly smaller than that of the bcc phase encountered in AlFeVSi systems (1.260nm). Also, the lattice parameter of the aluminum matrix was of 0.40461nm which is lower than the lattice parameter of pure aluminum (0.40494nm). After heat treatment, it was observed that the diffraction peaks intensity of the $\alpha$-Al$_{13}$(Fe,Nb)$_3$Si phase becomes higher, indicating a volume fraction increase of the silicide phase. Also, the aluminum matrix lattice parameter increase to 0.40475. There were other diffraction peaks that was not identified yet. The results suggest that the observed exothermic reaction arises from the precipitation of extra $\alpha$-Al$_{13}$(Fe,Nb)$_3$Si phase. Wang et al have reported the same precipitation phenomena in AlFeVSi alloys with Mm additions [12]. They attributed the $\alpha$-Al$_{13}$(Fe,Nb)$_3$Si precipitation to the formation of a metastable precursor Al$_8$Si$_4$Mn phase in the melt spun ribbons, which changes upon heating to the stable bcc silicides. Further investigations will be performed in the as-quenched ribbons in order to clarify their microstructures.

CONCLUSIONS

AlFeNbSi alloys ribbons were produced by melt spinning and their thermal stability and microstructures was investigated by means of DSC and x-ray diffraction. The conclusion are summarized as follow:
1) Nb addition is also very effective to stabilize the bcc $\alpha$-Al$_{13}$(Fe,Nb)$_3$Si silicide. However, its lattice parameters is smaller than that of the $\alpha$-Al$_{13}$(Fe,V)$_3$Si silicide in the AlFeVSi systems.

2) Unlike AlFeVSi systems in that systems it was observed a phase transformation in a temperature range of 400-490°C which was attributed to the extra-$\alpha$-Al$_{13}$(Fe,Nb)$_3$Si silicide.

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References