## SEM STUDIES ON THIN STRUCTURE SAMPLES OF AI-Ni EUTECTIC

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Abstract: Recently .Electron Beckscatter Diffraction -EBSD and Orientation Imaging Microscopy - OIM, have been shown to be useful in investigating the properties of thin materials samples of Al-Ni eutectic directional solidification of varying microstructures.

**Keywords:** SEM, EBSD, OIM, Eutectic, Solidification.

## 1. INTRODUCTION

The most common types of textures that were founded in metallic thin structure samples have been reviewed by Knorr [1]. It is a matter of experience, that thin structures with a high degree of (111) fibre texturing are desirable for interconnect applications.

The scientific literature identifies several reasons for desirability of a strong (111) fibre texture. It has been noted that (111) oriented grains remain primarily elastic during thermal cycling and are therefore more resistant to grain collapse as compared to non - (111) - oriented grains [2-4]. The presence of thermal hillocks has also been associated with grains lying away from the (111) orientation [5, 6]. This work describes the investigation on SEM - EBSD and OIM that have been undertaken at Physical - Metallurgy Laboratories F.S.I.M. - U.P.B., INAV-SA Bucharest, and D8 DISCOVER with GADDS -BRUKER AXS - Germany.

**2. EXPERIMENTAL RESULTS** Master ingots, with chemical composition given in Table 1, were elaborated in vacuum induction furnace by melting high - purity metals under argon atmosphere and were casting into graphite rods. These ingots were machined into rods and placed in high purity alumina crucible of  $47/57 \times 10^{-3}$  m inner/outside diameter [7]

Table 1. Chemical composition of eutectic Al-Ni

| Alloy     | <b>Ni</b> [at %] | <b>Al</b> [at %] |
|-----------|------------------|------------------|
| Al-5.7%Ni | 5.70             | Balance          |

Using OIM, several thousand measurements were made over the surface of the thin samples to

determine the texture of the material. Each was observed to possess a (111) fibre texture as seen by the pole figure representations shown in Fig 1. The thin structure samples were observed in Philips SEM 515 EDS, with an automated OIM attachment (TSL), [7]. Figure 2 shows a representation of an OIM scan with shading by image quality from a region of a specimen with lines of two different widths. The thick black lines of the image are the regions between metal lines. Grain boundaries are seen as black lines with those boundaries with a misorientation angle of  $5^{\circ}$  to  $15^{\circ}$  having thin lines and the boundaries with a misorientation angle greater than 15° as relatively thicker lines. Figure 3 shows some OIM images of the same region along with an additional patterned region with shading by lattice orientation. The grains which are within 5° of a (111) fibre texture are shaded dark, those with a misorientation of 5° to 10° a shaded somewhat lighter and those from  $10^{\circ}$  to  $15^{\circ}$  from a (111) fibre texture are shaded as the lightest shade of grey seen in the image. It is apparent from the images that the thin lines are generally all within 5° of the desired orientation, D8 DISCOVER with GADDS.

## 3. CONCLUSIONS

Substantial evidence has been obtained to suggest that textural evolution occur subsequent to patterning and post-patterning annealing. Little imagination is request to concept energetic arguments for why this structural evolution of thin structure samples occurs, especially for very thin line structures. As new surfaces are created, the structure seeks a more thermodynamically stable configuration. This often involves migration of grain boundaries as well as crystal lattice rotations. The most accurate method for showing such evolution is to map the local texture of a thin structure sample, which is subsequently patterned and annealed.

By re-mapping the same area as that initially measured, the comparison of the two structure maps would yield a direct correspondence and offer the evidence necessary to be assured that such evolution occurs. In absence of such an investigation, the assumptions are made that the initial local texture of the unpatterned and metal line areas was all initially identical and that the observed differences area function of the patterning.

This is likely a valid assumption as various regions of certain metalled wafers were tested for texture gradients without finding significant changes. In addition, the minor textural differences from position to position across a patterned structure between features of varying size were consistent over four different sets of measurements with the finer features exhibiting the strongest fibre textures. In general, the patterning of Al-Ni eutectic thin structure leads to a modification of the microstructure.

This is evidenced by both morphological rearrangement of the grain boundaries and by a strengthening of the crystallographic texture, with the texture of the finer patterned features being strengthened to the greatest extent. The GADDS allows very fast measurements in a few seconds or minutes, due to the sensitive 2D -detector.

Each diffraction image of the HI - STAR detector contains the information of many diffraction lines in a large angular range (details are given in this report)

Application like phase analysis, stress and texture measurements etc. can be performed with a high local resolution (micro - diffraction), since different sizes of small collimators (pinholes) are available.

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**Figure 1.** (III) pole figures of (a) strongly textured and (b) weakly textured thin structure corresponding to reliable and less reliable interconnects respectively







*Figure 3.* OIM generated images with shading by lattice orientation.