KANG et al.: GROWTH OF GRAINS DURING LIQUID PHASE SINTERING

layers grown during subsequent sintering cycles. Unfor-
tunately, most studies show that the Al₂O₃ spheres appear
to intrude into the Mo grains. From the etch bound-
aries near to the contact areas of Al₂O₃ spheres and Mo
grain it is obvious that the sintering consolidation is a
spatial effect which results from a fast deposition of material
onto Mo grains at the areas away from the contact
area and a negligible deposition rate at the contact
area itself.

By a modified contact flattening approach based
on a chemical potential increase at contact areas
caused by transmitted stresses and based on diffusion
of material through the thin liquid films in the contact
areas of Al₂O₃ spheres and Mo, the intrusion rate of
Al₂O₃ particles into Mo grains was calculated. It is
found to be orders of magnitude smaller than the
grain growth rate of Mo grains. The calculation and
the experiments both show that the shape changes of
grains due to contact flattening are negligible com-
pared to the shape change of grains resulting from
shape accommodation to the Al₂O₃ particles during
Owelding reopening.

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The main recrystallization texture components are the cube and R orientations. Their peak densities (Fig. 2) and volume fractions (Fig. 4) as well as the position of the skeleton lines (Fig. 3a) vary systematically with annealing temperature. As already demonstrated by pole figure data in [5], the intensity and volume fraction of the cube orientation are a minimum and those of the R orientation a maximum at 360°C. It will be seen also that for this temperature the recrystallization texture is quite similar to the rolling texture. Similarly the minor components and the background (BG) component show maxima at 360°C, although the latter is somewhat smaller than that for the as-rolled state.

For 400°C, the recrystallization texture is similar to that at 360°C except for a somewhat larger cube and smaller R component. For 520°C, in contrast, the annealing texture is composed of only two components, the R component and the Cube component with some characteristic distortions. For 280°C some characteristic differences in the recrystallization texture are found. The Cube component shows extremely strong scattering in the & g; direction, i.e. with respect to rotations around the rolling direction (Cubic & g; and Gost), and a component similar to the R recrystallization component is observed. It should be emphasized that none of these additional recrystallization orientations are found after annealing at 360°C (Table 1).

4. DISCUSSION OF THE ODF ANALYSIS

4.1. Decomposition of the ODF's into isotropic Gaus-type components

Although ODF's provide a much clearer insight into the orientation distribution than pole figures, two major difficulties are still encountered: (i) they are rather complex 3-dimensional functions which are difficult to visualize in Euler angle space with distorted metrics, (ii) they contain some grave errors (translation and ghost error) which are mainly due to the series expansion method used to reproduce the ODF's from the pole figures. For these reasons an approximation of the ODF's by model ODF's consisting of a superposition of isotropic Gaussian-type components has been applied here. This procedure which is justified by the excellent agreement between experimental and model ODF's (cf. Section 4.2) strongly reduces both difficulties and will now be demonstrated in detail. Some limitations of the model will be discussed in Section 4.3.

(a) In the form of volume fractions, M v of the components as given in Table 1 and Fig. 4 the method not only yields a simple, condensed and quantitative description of the texture but directly exhibits its principal features. It is superior to considering orientations involving only the peak heights f since it takes into account both the width c of the peaks (which strongly influences the volume fraction by the third power, and also their anisotropic scattering). This can be done by considering the deviation of the true position g of the component from the symmetric position and/or introducing auxiliary components (which may not result in a true maximum). Because of the minimization procedure the volume fractions

Fig. 2. (a-c) Approximate true (ghost corrected) ODF's for material rolled 90% (a) symbols indicate 40° c(111) rotated 5 components, and annealed 180 h at 280°C (b) 10 h at 360°C (c), 80 min at 400°C (d), and 60° at 520°C (e) V(110) = 22.

Fig. 3. (a) Path of, and (b) orientation density along, the skeleton lines (L(111) = 22). The paths given by the coordinates &phi; and &theta; of the intensity maxima in the sections &phi; = const; the dashed sections indicate ranges in which the path of the skeleton line changes strongly.
4.3 Anisotropic scattering

Because the scattering of the components is not usually fully isotropic measures must be introduced into the component analysis. However the calculation effort involved is such that only isotropic model functions can be applied.

(a) For the case of small anisotropy only the isotropic model component is used which, due to the minimization procedure, averages over the different directions. Errors appear in the difference ODF's, e.g. in Fig. 6(a) and 7(a) near the S position (φs, φ, φr = 68.4°, 30.4°, 50°) in the φ direction positive intensities appear (i.e. the model component is too small) and in the φ direction negative (shaded) intensities (i.e. the model component is too large). This indicates some preferred scattering the φ direction which is also found often for the R component, [except for high recrystallization temperatures, cf. Fig. 7(b)] and corresponds to a rotation around the sheet normal.

(b) If the anisotropy is too large, two overlapping isotropic components or auxiliary components can be introduced which might individually have no physical meaning. In such cases it is often useful to consider only the sum of their volume fractions in order to emphasize that physically they form only one component. An example will be given in (c) where two partial components are used to treat the Cube component.

(c) For a symmetric component anisotropic scattering can often be handled by placing the centre of the isotropic component not exactly at, but slightly off the origin, (e.g. the experimental (crosses) and model (circles) densities around the R position are plotted for the three directions corresponding to symmetries and positions arguments, but with no isotropic scattering. The amount and direction of this displacement (which can be determined once again by minimization) then provides a quantitative measure of the anisotropy. As an example the Cu position in Table 1 has been placed not at the symmetric position φl = 90°, but 20° which represents a slight rotation around the sheet normal. Since such effects are both frequent and reproducible, it must be assumed that such a shift has a physical meaning. Another example is apparent in Fig. 9 where the model component for this Cube position after recrystallization at 280°C occurs at φ = 83.8° and φr = 2° which describes a combined shift of 4.2° around the rolling direction and of 2° around the transverse direction.

(d) The Cube position often shows systematic scattering which is sometimes rather large. After recrystallization at 520°C Fig. 8(c), for example, scattering is observed in the φ, and φ directions corresponding to rotations around the normal and the rolling direction. The measures described in (b) and (c) must then be applied simultaneously in order to obtain the intensity distribution. In the present text the Cube model consists of two components (Cube1 and Cube2, Table 1). Figure 10(b) which shows the true model functions ftrue(φ) in the plane φr = 0° demonstrates the structure of the peak. The full curves give density lines for the individual components and the dashed lines the superimposed densities. It will be apparent that the complicated structural features of the peak, namely extension in the φ direction close to the Cube position with a high intensity level and in the φ direction further away from it with a low level, are very well reproduced by the two types of Gausss components. It will be seen also from Fig. 10(a) and (d), that the quantitative agreement with experiment is excellent.

5. DISCUSSION OF THE RECRYSTALLIZATION BEHAVIOUR

5.1. Recrystallization of the dilute Al-Fe alloys

In the recrystallization textures of rolled pure Fe-Cr with high stacking fault energy the Cube orientation is the dominant texture component and in the particular case of high purity aluminium only this component appears. Small additions of foreign atoms often lead, however, to a second texture component, the R orientation, as in the case of an alloy with only 0.004% Fe, solid in cold solution [6]. Since the R component is hardly observed in other materials, it can be assumed that its formation is related to the strong S orientation typical of the rolling texture of Al. According to Beck [1] the R component can be formed from the S component in two ways: (i) by continuous recrystallization, since both orientations, S and R, are rather similar, and (ii) by discontinuous recrystallization, since 40° (111) orientation relationships exist between symmetrically equivalent variants of S and R which lead to high growth rates [1, 18, 19, 20].

Hirsch and Lücke [5, 6] investigated the recrystallization textures of dilute Fe-Al alloys after systematic variation of the preannealing conditions, Fe content and recrystallization temperature and were able to analyze the rather complex recrystallization behaviour on the basis of the precipitation state of the iron which has a very low solubility in A1. They observed, amongst other things, the Cube component minimum near 360°C (Fig. 4) and concluded that because of the boundaries were strongly pinned by precipitation during recrystallization, continuous recrystallization was the dominant process at this temperature. At higher annealing temperatures discontinuous recrystallization predominated. These unexpected findings will now be checked by a detailed analysis of the present ODF's. The physical reasons for such behaviour have been discussed in [6] and will not be treated here.

5.2. Evidence for continuous recrystallization

At 360°C the recrystallization texture is almost identical to the rolling texture. This is shown in Fig. 1(a), c) for the pole figures, Fig. 2(a), c) for the orientation densities, Fig. 3(a); b) for the position and density of the skeleton line, Fig. 4 for the volume fraction of major and minor components and in Table 1 for the orientations of the main components. The background (BG) has decreased from 25% after rolling to 14% after recrystallization at
precise texture analysis of this type yields information not previously available, e.g., the volume fractions of the individual texture components, the observation of small changes in the various theories (even if the corresponding maxima are hidden) and the anisotropy of their scattering.

These new data provide additional aids in clarifying the underlying mechanisms of recrystallization. Thus in the present work it has been possible for the first time to clearly distinguish between the separate processes of continuous (in situ) and discontinuous (pseudo) recrystallization even after recrystallization was completed and to assess the quantitative contribution of each process.

In addition more precise details about the effects of oriented nucleation and oriented growth on the formation of the Cube and R orientations during discontinuous recrystallization have been obtained. Knowledge of the exact position of the R component and the direction of scattering of the Cube component has shown that at low deformation temperatures (280°C) the texture is determined mainly by nucleation processes whereas at high temperatures (350°C) selective growth of the new Cube and R grains is mainly responsible for their dominance. This is probably due to the suppression of oriented growth by the precipitation of the FeS phase. The formation of the Cube before recrystallization starts here, whereas at high temperatures (350°C) the FeS is completely in solution.

Determination of the respective volume fractions at intermediate temperatures (300°C) has shown that recrystallization in situ is the dominant process. This can be attributed to precipitation during recrystallization which is known to impact on textures at low temperatures.

An examination of the deformation and recrystallization processes in these alloys revealed that a low FeS content very effectively (Ito and Lücke [5]). The above results obtained by quantitative texture analysis have greatly increased understanding of the recrystallization processes in these alloys.

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INTRODUCTION
Several years ago, a technique [1, 2] was developed for producing binary alloy thin films containing short-wavelength (8.8–10 nm) composition modulations. It is well known that such a layered structure exhibits characteristic X-ray patterns with satellite reflections around the Bragg peak. The course of diffusion in modulated samples can be followed from studying the progressive intensity change of the satellite as a function of annealing time. Hilliard and his co-workers [3, 4] utilized such a technique successfully to check the modified continuous diffusion equation, which contains additional terms introduced by

Hillier [5] and Cahn [6] and is the basis of the theory of spinodal decomposition. More recently, Cook et al. [7] derived a discrete model for the diffusion equation. In a more fundamental approach utilizing the kinetics of the phase model, Yamashita [8] and Khachatryan [9] presented various theoretical models of order–disorder kinetics in a unified manner. Tsakalakos [10, 13] observed a minimum in the diffusivity of Cu–Ni with a modulation length at a wavelength of about 1.5 nm which could not be explained by either Cahn's continuum model or Hilliard's discrete model. Hence, the discrete model leads to a nonlinear theory of diffusion which explains successfully the stability and kinetics of the concentration waves.

Recently, there are increasing interests in studying similar diffusional effects in ternary alloys. It is thus

MEASUREMENTS OF INTERDIFFUSIVITIES IN CuNiFe TERNARY ALLOY THIN FILMS
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Abstract—Interdiffusivities have been measured in CuNiFe ternary alloys containing 53 at. % Cu, 40 at. % Ni, and 7 at. % Fe. Isothermal anneals were performed at temperatures 320, 345 and 400°C using thin films containing composition modulations (8.8–10 nm) generated by the vapor-phase growth technique. Spinodal decomposition in these films was observed in the X-ray diffraction pattern as indicated by the growth of satellite intensities at 320°C. The diffusivities at each temperature were determined from the growth or decay rate of the composition modulation. A plot of the interdiffusion coefficient Dc versus the dispersion relation of the spinodal wave determined by X-ray diffraction analysis of the CuNiFe ternary alloy is presented.

Résumé—Nous avons mesuré les interdiffusivité dans des alliages ternaires CuNiFe contenant 53 at. % Cu, 40 at. % Ni, et 7 at. % Fe. Nous avons effectué des isothermes de traitement thermique à des températures de 320, 345 et 400°C, en utilisant des films minces contenant des modulations de composition (8.8–10 nm) produites par la technique de croissance en phase vapeur. Nous avons observé la décomposition spinodale dans ces films par le diagramme de diffraction de rayons X qui mettait en évidence une croissance d'intensité des satellites à 320°C. Nous avons mesuré les diffusivité à chaque température à partir de la vitesse de croissance ou de décroissance de la modulation de composition. Le coefficient d'interdiffusion Dc en fonction de la relation de dispersion de spinodale a été mesuré par la diffusion des rayons X avec une croissance anormale pour certaines longueurs d'onde pour lesquelles on observait un accroissement du module élastique. Nous avons évalué la température spinodale critique et nous avons calculé les trois premiers coefficients de l'énergie libre à l'aide d'un ajustement par regresion non linéaire de la courbe de Dc en fonction de βs.


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