Texture mechanism in some fcc-type soft magnetic materials

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The authors have studied the mechanism for recrystallization texture in some soft magnetic materials with fcc crystal structure. The alloys used were 77% Ni–14% Fe–5% Cu–4 wt % Mo permalloys. Thin foils selected area electron diffraction (SAD) and X-ray diffraction techniques were employed using a Philips 300 Electron Microscope (EM 300) and an X-ray diffractometer, respectively. Investigations were carried out on deformed, recovered and recrystallized states of the alloys possess copper-type of rolling texture with $\{110\}\langle 112\rangle$ texture as the predominant deformation texture though other minor components such as $\{112\}\langle 111\rangle$, $\{110\}\langle 001\rangle$ and $\{123\}\langle 420\rangle$ textures were detected. No cube texture, $\{100\}\langle 001\rangle$ was detected in any of the deformed and recovered materials though the recrystallization texture in these alloys is the cube texture, $\{100\}\langle 001\rangle$ which forms over 80% of the annealing texture in these alloys.

It is concluded here that the detection of cube texture in the deformed and recovered materials is not a prerequisite for the detection of cube texture in these alloys. The present work is not conclusive about the mechanism for recrystallization texture, but it is proposed here that recrystallization texture, in these alloys is attributed to the growth-oriented mechanism based on the following model. (1) The lattice domains which form the recrystallization texture are present in the cold-rolled matrix. (2) The favoured site for nucleation are the grain boundaries and deformation band boundaries. (3) For the nucleus to be able to grow and form the recrystallization texture it must possess the necessary free energy. (4) Grains must be capable of growth into two or more orientations between which it forms, i.e. the nuclei which form the cube texture should have a [1 1 1] pole in common with the matrix in which they grow and a rotation of about 30° around this pole.

1. Introduction

The curiosity of the human mind to find the origin of things continues to grow daily unabated perhaps because of scientific adventures or applications, philosophical or religious interests or needs. The origin of recrystallization textures and their effects on finished products in fcc metals and alloys have been of immense importance to various early researchers [2–8].

Other workers have also studied the origin and effect of recrystallization textures on magnetic properties in some soft magnetic materials [3, 8-11]. Various factors such as the presence of nucleating agents, composition, grain sizes at various stages of working and annealing schedules, working schedules, and the annealing atmosphere are known to affect recrystallization texture.

The resulting texture in these materials due to these factors could be simple, and sharply defined, or complex, or even with many components with a nearly random distribution of grain. In most cases, the cube texture, $\{100\}\langle 001\rangle$ which forms more than 90% of the annealing texture in these materials is usually described as the annealing texture, though other tex-

tures such as $\{358\}\langle 835\rangle$, $\{113\}\langle 875\rangle$, annealing twins, etc. have been reported [2–4].

Two schools of thought have emerged from previous works (the oriented-growth and orientednucleation mechanisms) to explain the fundamental cause of annealing texture in fcc metals and alloys. Though both theories have one or other deficiency, they have one important assumption in common, that is, the lattice domains that form the cube texture after recrystallization are already present, as a minor component, in the cold-rolled matrix from which the cube texture emerges. Arguments for and against have been presented in the literature for both theories [2, 4, 11, 12].

The work reported here forms part of a more general investigation of the magnetic properties, microstructure and magnetic domain structure of 77% Ni permalloys [1].

2. Materials and experimental techniques

The materials used throughout the entire investigation had an approximate percentage weight composi-

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tion 77 Ni–14 Fe–5 Cu–4 Mo supplied in the form of 375 μ m thickness cold-rolled sheets, with the trade name "Nilomag 77". The alloys have fc c crystal structure at stoichiometric composition Ni₃Fe. Each material contained varying quantities of added and impurity elements such as Mn, Mg, O, S, C, N, etc. The main aim of the investigation was to study a mechanism for recrystallization textures in these alloys, consequently, cold-rolled (deformed), recovered and recrystallized materials were used. Two methods were used to thoroughly investigate the recrystallization texture mechanisms in these alloys.

Firstly, a Philips 300 electron microscope (EM 300) employing thin foils and selected area electron diffraction techniques were used. Over 300 diffraction patterns were taken of different areas of the cold-rolled and recovered materials, while over 200 diffraction patterns were taken of different areas of the recrystallized materials. Secondly, X-ray techniques using a texture goniometer were used to monitor texture formations in these alloys. The specimens used were cut to approximately 25 mm square. A layer of about 30 µm was removed by etching with a carapellar reagent. This ensured that surface defects were removed. The texture was determined using a texture goniometer set to receive the $\{1 \ 1 \ 1\}$ reflections. The equipment consisted of a stabilized X-ray generator using a copper source to produce CuK_{α} radiation, a goniometer, a scintillating counter, electronic circuitry, and a chart recorder. An accelerating voltage of 30 kV and a current of 10 mA were used. The plot from the chart recorder was transferred on to a $\{111\}$ pole figure.

The recovered materials were annealed at $550 \,^{\circ}$ C for varying annealing periods of 5, 10, 15, 20, 30, 40, 60, 85 and 120 min, and 800 to 1200 $^{\circ}$ C for annealed recrystallized materials.

3. Results and discussion

As stated previously, thin foils and selected area electron diffraction techniques (SAD) were employed using a Philips 300 electron microscope (EM 300), while a texture goniometer was used for texture determinations on the cold-rolled, recovered and recrystallized materials.

By cold-rolled materials, we mean materials that have undergone cold-work. These materials have very high dislocation density due to the varying degrees of plastic deformation caused by mechanical strains. In the case of recovered materials, the distribution and density of defects in these materials have changed. On the other hand, the process of recrystallization involves "nucleation" and "growth" processes in which subsequent growth occurs through the deformed material on the site where a stable nucleus was formed. Primary recrystallization is the process in which new grains are nucleated and these then grow at the expense of the deformed matrix until this is all consumed.

Over 300 diffraction patterns of different areas of the cold-rolled and recovered materials were obtained.

Figs 1 to 3 show some of the micrographs obtained for the deformed and recovered materials.

The $\{110\}\langle 112\rangle$ texture which is usually associated with alloys of low stacking fault energy (or more precisely low γ/Gb) was found to be the predominant deformation texture though other minor components such as $\{112\}\langle 111\rangle$, $\{110\}\langle 001\rangle$, $\{123\}\langle 420\rangle$ etc. textures were also detected. No cube texture was observed from all the diffraction patterns taken from the deformed and recovered materials. The reasons for the non-detection of the cube texture in the cold-rolled and recovered materials [1] could be due to the following facts.

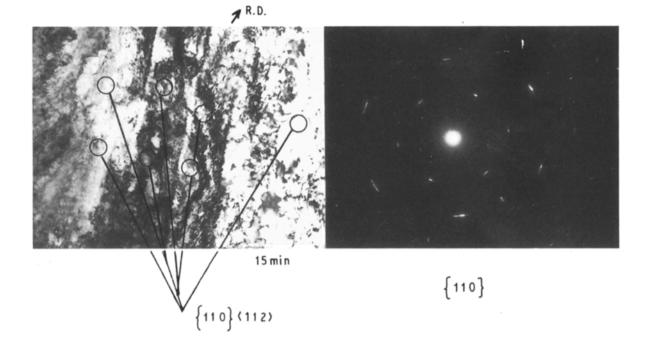
(1) At this stage the nuclei for the cube texture might be too small (i.e. the size could be less than the critical size needed for detection).

(2) The crystals might not have been sufficiently rotated away from their cold-rolled directions.

From Fig. 3 it can be seen that there were growths of new strain-free grains which started at many different locations. The non-detection of cube texture in the cold-rolled and recovered materials were further confirmed by Fig. 4 which shows the $\{1\ 1\ 1\}$ pole figure for a cold-rolled material. From this figure, one could easily see the strong $\{1\ 1\ 0\} \langle 1\ 1\ 2\rangle$ and $\{1\ 1\ 2\} \langle 1\ 1\ 1\rangle$ deformation textures in the cold-rolled material. These results are in good agreement with the deformation textures observed previously in some f c c metals and alloys [14–16]. These alloys have copper-type deformation texture.

It was also noted that as the annealing temperatures increased there was further grain growth in addition to primary recrystallization and this resulted in the development of cube texture, $\{100\}\langle 001 \rangle$. Figs 5 and 6 are the $\{111\}$ pole figures showing the development of cube texture $\{100\}\langle 001 \rangle$ as the annealing temperature is increased. Fig. 5 is the $\{111\}$ pole figure showing the slightly developed cube texture $\{100\}\langle 001 \rangle$ after annealing the materials at 900 °C for 4 h and furnace-cooled to room temperature.

In Fig. 6, the $\{111\}$ pole figure shows a more strongly developed optimum cube texture $\{100\}\langle 001\rangle$ and weaker $\{146\}\langle 211\rangle$ and $\{123\}\langle 420\rangle$ retained deformation textures after an anneal of 4 h at 1100 °C. These results suggest that the cube texture is not merely a primary recrystallization texture but also a grain growth texture, that is, primary recrystallization with different orientations can occur, the cube orientation then grows. In these alloys, as the annealing temperature is increased above 1100 °C secondary recrystallization took place in the material which destroyed the cube texture. The reason is that as the high angle boundaries continue to sweep through the material, they also cause the grains with which they come in contact to experience rotation which consequently causes some grains to rotate away from the $\langle 001 \rangle$ direction. It was also known [10] that as the degree of cold-work or deformation increases, the $\{110\}\langle 112\rangle$ deformation texture and the cube texture $\{100\}\langle 001\rangle$ after recrystallization increase though the retained deformation texture decreased. The cold-work will increase the deformation band boundaries (the nucleation sites) which then results in high intensity cube



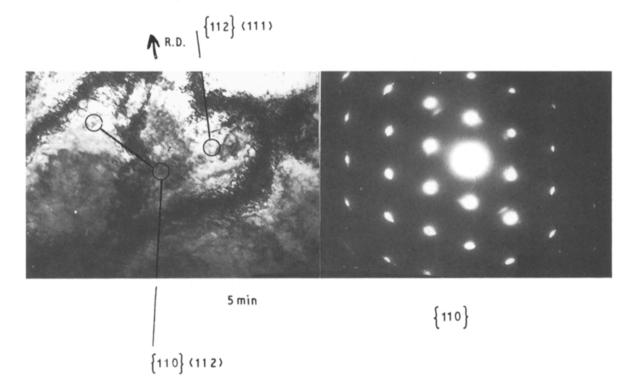
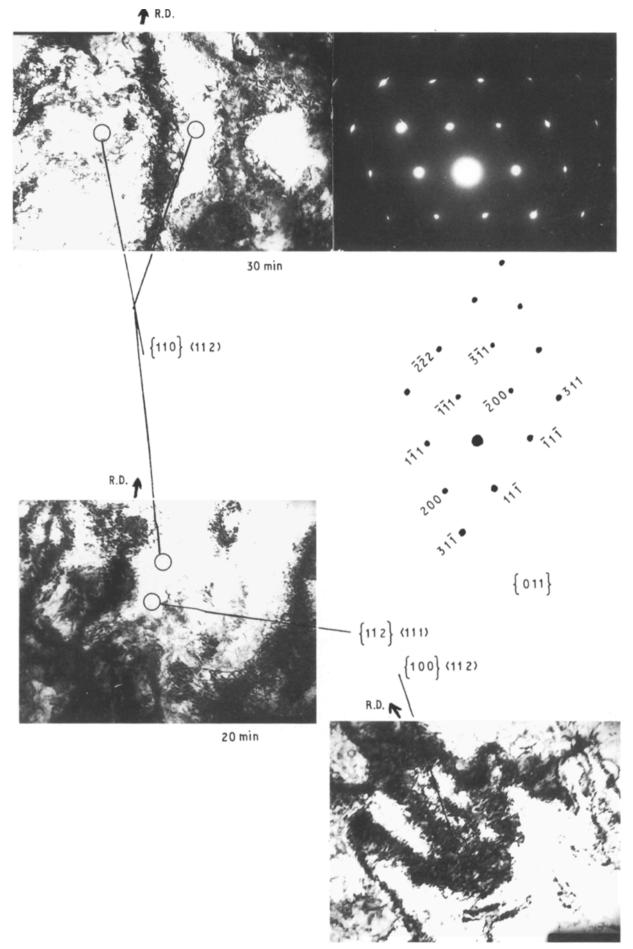


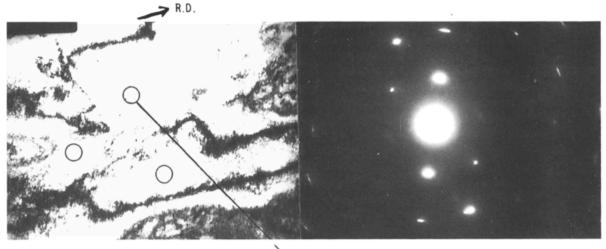
Figure 1 Diffraction patterns of a recovered material annealed at 500 °C for 5 min showing strong $\{110\} \langle 112 \rangle$ deformation texture. (RD = rolling direction).

texture during primary recrystallization. It is also known that cold-work will increase the stored free energy in the material. This is the energy which provides the driving force for the embryos to grow to their critical size before final growth and reorientation take place by the nuclei during primary recrystallization. Two theories (the oriented-growth and orientednucleation) have been put forward to explain the mechanism for the formation of primary recrystallization textures in fcc metals and alloys. Both theories are known to have one important assumption in common namely that the cube lattice domains that form

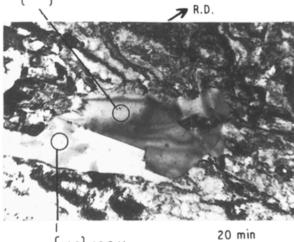


10 min

Figure 2 Diffraction patterns of recovered materials annealed at 500 °C for 30 min showing deformation bands. $\{110\} \langle 112 \rangle$ texture was observed both within and outside the bands.

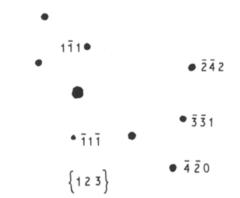


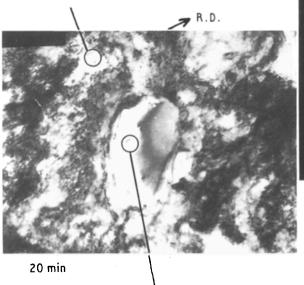
{110} (112)

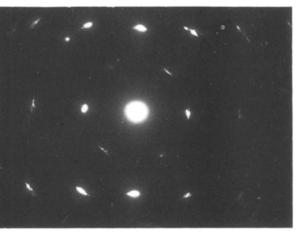


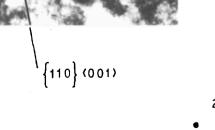
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{110} (001)









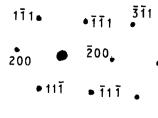


Figure 3 Micrographs of recovered materials annealed at 550 °C for 20 min showing strong $\{110\}\langle 001 \rangle$ and $\{123\}\langle 420 \rangle$ deformation textures. No cube texture $\{100\}\langle 001 \rangle$ was observed. Note the formation of recrystallized strain-free grains at different sites.

{110}

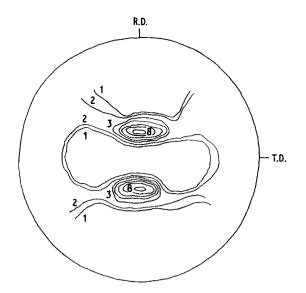


Figure 4 {111} pole figure showing strong $\{110\}\langle 112\rangle$ and $\{112\}\langle 111\rangle$ deformation textures in a cold-rolled material of 375 µm thickness.

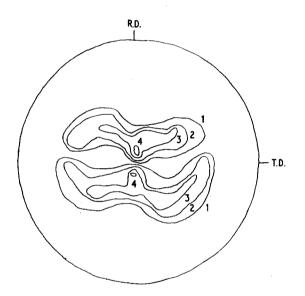


Figure 5 {111} pole figure showing the slightly developed cube texture $\{100\}\langle 001 \rangle$ after an anneal of 4 h at 900 °C.

the cube texture after recrystallization are already present, as a minor component, in the cold-rolled matrix from which the cube texture emerges. Arguments for and against have been presented in the literature for both theories. From the present results, it is hereby proposed that though oriented-growth mechanism is the most likely mechanism for recrystallization texture in these alloys, more work needs to be done to be conclusive.

Nucleation is defined [17] as "the initiation of transformation at discrete sites in the parent phase", while "the subsequent advance of the transformation product at the expense of the parent phase is regarded as growth".

In supporting any of the theories on mechanisms for recrystallization texture in these alloys, one needs to ask questions such as whether the orientations absent from (or only weakly represented in) the recrystallization texture are suppressed because of the unavail-

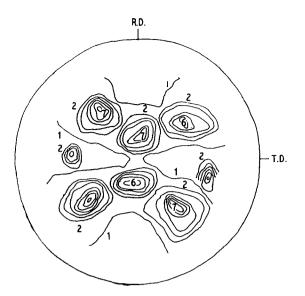


Figure 6 {111} pole figure showing a more strongly developed cube texture $\{001\} \langle 001 \rangle$ and strong deformation textures $\{146\} \langle 211 \rangle$ and $\{123\} \langle 412 \rangle$ after an anneal of 4 h at 1100 °C.

ability of nuclei in these orientations (oriented-nucleation mechanism) or because of the inability of such nuclei to grow to an appreciable volume in competition with nuclei of other orientations (oriented-growth mechanism).

While supporting the growth-oriented theory, it is hereby suggested that since none of these theories individually accounts for the various experimental observations, a composite theory which combines the successful points of each theory would be needed to satisfactorily explain the mechanism for the formation of primary recrystallization texture. This proposes a new theory based on four principles.

(1) The lattice domains which form the recrystallization texture are present in the cold-rolled matrix.

(2) These domains should possess a minimum amount of free stored energy which will allow them to grow to a critical size during the nucleation period.

(3) There should be suitable lattice sites at which the lattice domains should be able to grow initially to a critical size. After this initial growth to a critical size, the likely lattice sites which include grain boundaries and deformation band boundaries (which are known to favour rapid development of the rolling texture to annealing texture) should be available.

(4) After attaining the critical size, the domains should be capable of growing into two or more orientations between which it forms.

When a nucleus at the embryo or nucleation stage possesses the required orientation for growth but not the required amount of free energy it will never grow to its critical size and vice versa, consequently, it will be consumed by other nuclei that possess the required free energy and orientation. After attaining the critical size, the nucleus should be able to grow if there are lattice sites such as grain boundaries and deformation band boundaries to grow into. The nuclei that are then favoured to grow to the annealing texture or the cube texture, $\{100\}\langle 001 \rangle$ should have the $\{111\}$ pole in common with the matrix in which it grows and a rotation of about 30° around this pole. The individual orientations in the spread should have one of the four symmetrical components derived from two $\{110\}\langle 112\rangle$ components by rotation of about 30° to give $\{110\}\langle 112\rangle \pm 30^\circ$, $\{110\}\langle 1\bar{1}2\rangle \pm 30^\circ$. Finally, the nucleus, which is capable of growth into two orientations, will be able to form an annealing texture while any nucleus which is capable of growth into the four orientations will form the predominant annealing texture.

Violation of any of these models will result in the cold-rolled texture being consumed by other textures at the latter stage of recrystallization.

While supporting the oriented nucleation theory [4] invoked the process of polygonization, Burgers indicated that for this process to be operative, there could be cube components in the cold-rolled matrix. Burgers [4], therefore, went further by assuming that before the actual growth starts, the process sufficiently frees for growth (not further explained by Burgers) the cube-oriented domains in the cold-rolled texture from dislocations, so that they are able to grow at the expense of their surroundings and thus serve as nuclei for the recrystallized state. Grewen and Huber [18] with work on 30% Ni-Fe alloys detected cube texture in the cold-rolled matrix. They, like Burgers concluded that the necessary condition for the formation of cube texture during primary recrystallization is the presence of cube texture in the cold-rolled material. Grewen and Huber also concluded that the nucleation-mechanism of the cube texture cannot be finally explained on the basis of their results. The results reported here contradict previous findings [4, 18], because there was no cube texture detected in the deformed (cold-rolled) and recovered materials, hence it could be concluded here that the presence of cubeoriented domains in the deformed matrix is not a prerequisite for the formation of cube texture as the recrystallization texture in these alloys. This conclusion is in accordance with the findings of Schmidt et al. [7]. Though Verbraak [19] supported the oriented-nucleation theory by the Rowland transformation, in this way a $\{1 \ 1 \ 2\} \langle 1 \ 1 \ 1\rangle$ oriented twinned domain present in the rolling texture could be transformed "as an entity into a cube-oriented domain", he also found that cube texture could form in an annealed or recrystallized material even if there was no cube texture in the original cold-rolled matrix. One of the difficulties of the so-called oriented-nucleation theory is that the primary atomic movements which give rise to the formation of a nucleus most probably occur in regions with dimensions smaller than can be presently resolved by the usual method of transmission microscopy (with the possible exception of the field-ion microscope and most data on nucleation have been deduced from a later stage of the growth of the crystallites. Cohen [17] referred to it as "operational nucleation" which is quite different from the "theoretical nucleation", and he, therefore, concluded that any evidence from such data would be susceptible to various interpretations of what actually happens in the true nucleation process and any conclusions derived from it may be ambiguous and misleading.

Dillamore [2] has supported the concept of orientedgrowth mechanism and set up a model to relate annealing textures to the deformation texture from which they form. This model was based on seven principles: the definition of fcc deformation textures, the likely sites for nucleation which are grain boundaries and deformation band boundaries, and the ability of the nucleus to grow through a big spread about the orientations of its parent components so that the nucleus forms part of the annealing texture. Dillamore explicitly stated that apart from the orientation required by the oriented-growth theory for easy boundary displacement, the nucleus should have "the ability" (not further defined) to grow into its surroundings. Could this "ability" be expressed as the process which occurs at the atomic level? Secondly, Dillamore [2] has not fully explained the process by which the nuclei are formed. Despite the above deficiencies which Dillamore's theory has failed to resolve, this theory could be easily seen to be based on the idea of selective growth in which the grains which satisfy the orientation relations and having the ability (probably required free energy) to grow into their surroundings do so at the expense of their neighbours. Lucke [20] noted that the same rolling texture in high-purity copper rolled at temperatures between room temperature and - 125 °C produced markedly differing recrystallization textures. Lucke then concluded that the difference cannot be adequately explained purely on the basis of selective growth but must be connected with the process of nucleation.

It is again evident [2, 19-22] that the growthoriented hypothesis based on selective growth is still very inadequate to explain recrystallization textures in fcc metals and alloys.

4. Conclusions

Recrystallization texture mechanisms in some fcctype soft magnetic alloys have been studied. The $\{110\}\langle 112 \rangle$ deformation texture was found to be predominant, though deformation textures such as $\{112\}\langle 111 \rangle$, $\{110\}\langle 001 \rangle$ and $\{123\}\langle 420 \rangle$ were detected. No cube texture was detected in the coldrolled and recovered materials, even though the recrystallization texture was the cube texture, $\{100\}$ $\langle 001 \rangle$ which formed more than 80% of the annealing texture in these materials.

It is proposed here that the mechanism for recrystallization or cube texture in these alloys is the growth-oriented mechanism based on the following model.

(1) The lattice domains which form the recrystallization texture are present in the cold-rolled matrix.

(2) The domains should possess a minimum amount of free stored energy which will allow them to grow to a critical size during the nucleation period.

(3) There should be suitable lattice sites at which the lattice domains should be able to grow initially to a critical size. The likely sites are grain boundaries and deformation band boundaries.

(4) After attaining a critical size the grains should be capable of growing into two or more orientations

between which it forms. The nuclei which form the cube texture should have a $\{1\,1\,1\}$ pole in common with the matrix in which they grow and a rotation of about 30° around this pole.

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