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The influence of texture on the magnetic permeability of 77 wt% Ni permalloys

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The effect of annealing on the initial magnetic permeability of cold-worked permalloys, of approximate wt% composition 77 Ni, 14 Fe, 5 Cu and 4 Mo, has been investigated and interpreted in terms of the development of crystallographic texture. Two competing recrystallization textures have been observed. One is a modification of the deformation texture, with a range of components between $\{1\,1\,0\}\langle 1\,\bar{1}\,2\rangle$ and $\{1\,1\,2\}\langle 1\,1\,\bar{1}\rangle$, and the other is the cube texture. The two textures develop approximately equally in the temperature range from about 950 to 1050° C, but the cube texture is dominant after annealing between about 1050 and 1100° C. In material annealed for 4 h the magnetic permeability increases to a maximum after annealing at about 900 to 950° C, due to primary recrystallization, and then decreases to a minimum at about 1050° C, before rising to a maximum at about 1100° C and decreasing at higher temperatures. It is thought that the retained deformation texture tends to decrease the permeability and this accounts for the decrease above about 900° C. However, the cube texture increases the permeability and when it becomes dominant above 1050° C, the permeability increases. Secondary recrystallization above about 1100° C destroys the texture and the permeability is reduced. Similar effects on the magnetostriction constant have been observed.

1. Introduction

The Ni–Fe permalloys are widely used commercially because of their very high initial magnetic permeabilities. They are also susceptible to magnetic annealing, which enables the shape of the hysteresis loop to be tailored to device requirements. Magnetic properties can be dependent upon the crystallographic texture produced by forming and heat-treatment schedules, as well as on the presence of short and long-range order, particularly in alloys close to Ni_3Fe [1–5]. The achievement of optimum magnetic properties depends on having low oxygen, sulphur and carbon contents, and the permalloys are usually given a high-temperature anneal in hydrogen to lower the concentration of these harmful interstitials.

The permalloys used in this work had wt % compositions close to 77 Ni, 14 Fe, 5 Cu and 4 Mo. In these alloys, the additions of molybdenum and copper enable high initial permeabilities to be achieved when a critical degree of short-range order is produced during a low-temperature anneal or a furnace cool [6]. This critical amount of short-range order gives approximately zero values of the anisotropy and magnetostriction constants. This paper reports the results of an investigation of texture formation in cold-rolled and in annealed material, and the effect of texture on the initial permeability and magnetostriction constant. It forms part of a more general investigation of the magnetic properties, microstructure and domain structure of 77% Ni permalloys [7].

2. Materials and experimental technique

The alloys were commercially produced with approximate wt % composition 77 Ni, 14 Fe, 5 Cu and 4 Mo. Table I gives the compositions of the alloys, the balance being nickel with typically 0.2 wt % of impurities, the main impurities being chromium, cobalt and magnesium. Manganese is present in the alloys to improve hot-workability. The materials were supplied in the form of 375 μm cold-rolled sheet.

Initial permeability measurements were made on specimens in the form of annular rings of 17.5 mm inside diameter and 25.4 mm outside diameter, punched from the sheet material. Annealing was carried out in a purified, dried hydrogen atmosphere, followed by a furnace cool. The permeability was calculated from the measured inductance at 1592 Hz of a toroid wound around five rings, as described elsewhere [6, 7].

Texture formation was investigated in the alloys in the cold-rolled condition and after annealing in purified dry hydrogen between 900 and 1250° C for periods from 2 to 6 h. Specimens approximately 25 mm square were used and the texture was determined using a texture goniometer set to receive the $\{1\,1\,1\}$ reflections, the radiation used being Cu$K\alpha$. The chart recordings from the texture goniometer were used to construct $\{1\,1\,1\}$ pole figures. A Philips EM300 electron microscope was used to investigate the initial stages of the formation of the recrystallization texture,

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TABLE I Composition of the alloys (wt %)

Alloy	Mo	Cu	Fe	Mn
A	3.96	4.86	13.9	0.67
B	4.16	5.11	13.4	0.77
C	4.18	4.94	13.4	0.71
D	4.18	4.96	13.6	0.66
E	4.27	5.24	13.5	0.72

using cold-rolled material heat-treated between 300 and 550° C for times for 5 min to 2 h.

Some magnetostriction measurements were carried out on material annealed between 600 and 1200° C. The technique has been described previously [6].

3. Results and discussion

Fig. 1 is a $\{111\}$ pole figure for the cold-rolled material. This deformation texture may be interpreted in terms of a range of components between $\{110\}\langle 1\bar{1}2\rangle$ and $\{112\}\langle 11\bar{1}\rangle$, with intermediate orientations such as $\{123\}\langle 41\bar{2}\rangle$. This texture has been observed by other workers in similar fcc metals and alloys. Selected-area diffraction by transmission electron microscopy also gave similar results. No cube texture was detected by X-ray or electron diffraction in the cold-rolled and recovered materials. The apparent lack of cube-oriented domains in the material observed in the electron microscope could have been due to inability to observe a large enough area of foil.

Figs 2 to 6 show the effect of heat treatment on texture formation. Fig. 2 shows the $\{111\}$ pole figure for a material annealed at 900° C for 4 h. At this temperature primary recrystallization is complete and, as well as a weak remnant of the deformation texture, a weak cube texture $\{100\}\langle 001\rangle$ has developed. Figs 3, 4 and 5 show the approximately equal development of the retained deformation texture and the cube texture after 4 h anneals at increasing temperatures up to 1050° C. Development of the two competing textures with time at 1100° C is shown in Figs 6, 7 and 8. The retained deformation texture at first develops more rapidly than the cube texture and is the stronger of the two after a two-hour anneal. At longer times the retained deformation texture weakens relative to the cube texture, which is the dominant texture after 6 h. It can be seen from Fig. 7 that at 1100° C minor components of the retained deformation texture have developed preferentially. This behaviour with annealing time suggests that if we consider a standard 4 h anneal, the cube texture will start to be the dominant texture above about 1050° C. Fig. 9 shows that above 1100° C both textures are weaker, and this was found from optical and electron microscopy to be due to rapid secondary recrystallization.

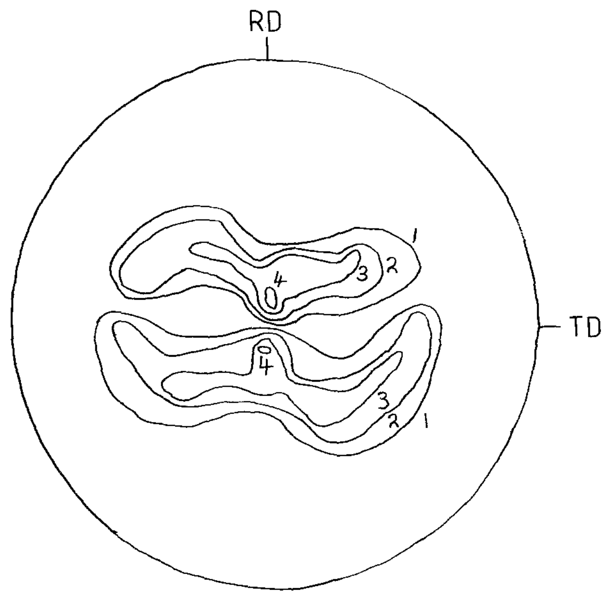

Figure 2 $\{111\}$ pole figure for material annealed for 4 h at 900° C.

The textures developed in the same manner for all the alloys, but it appeared that the strengths of both the deformation and the recrystallization texture increased with molybdenum concentration.

The effect of the amount of prior deformation on the development of the annealing texture was investigated by cold-rolling the original sheet of 375 μm thickness. The results showed that the strength of the

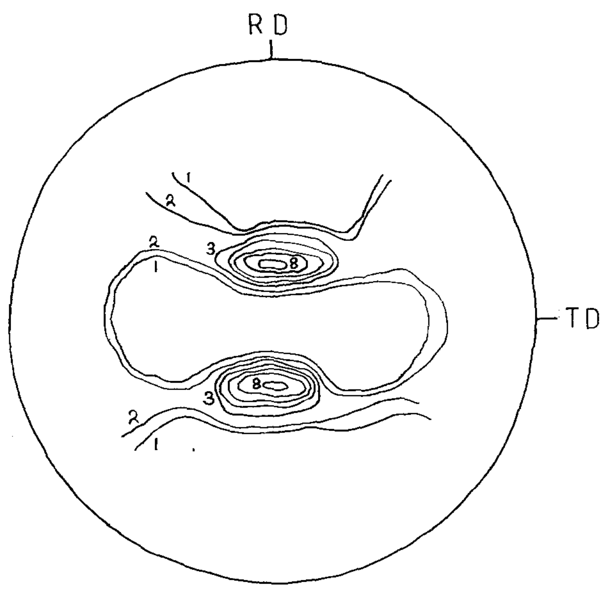

Figure 1 $\{111\}$ pole figure of cold-rolled material.

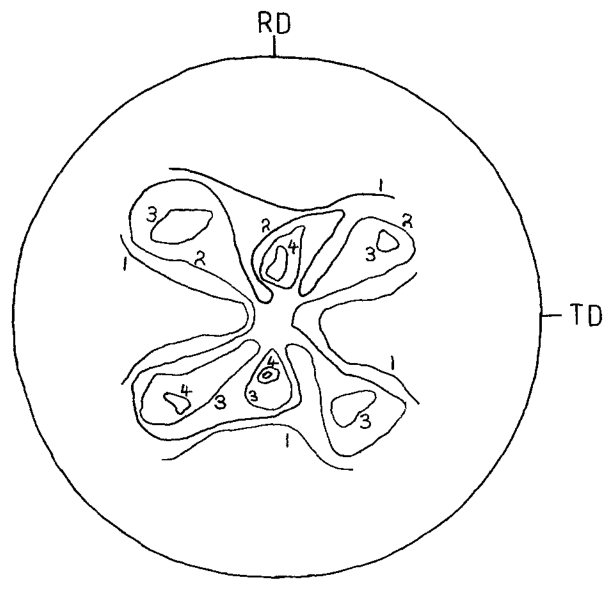

Figure 3 $\{111\}$ pole figure for material annealed for 4 h at 950° C.

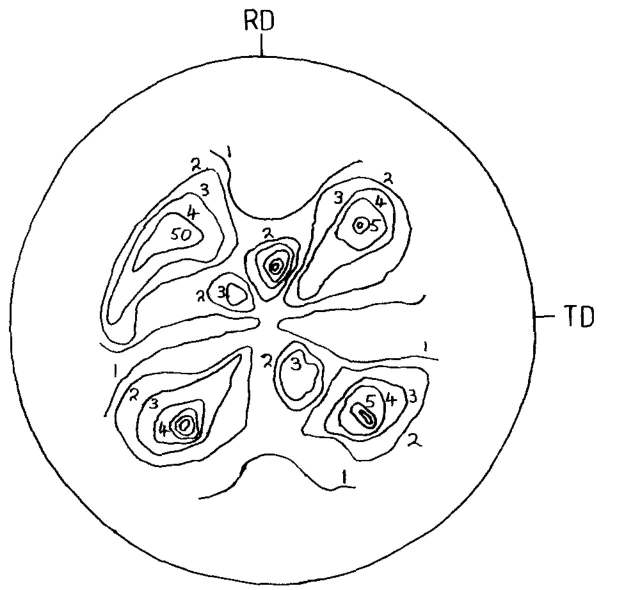

Figure 4 {1 1 1} pole figure for material annealed for 4 h at 1000° C.

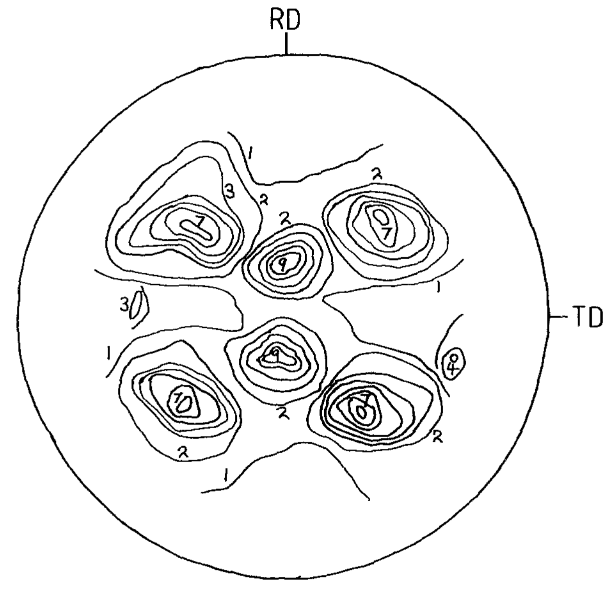

Figure 6 {1 1 1} pole figure for material annealed for 2 h at 1100° C.

cube texture increased with increasing deformation, whereas the retained deformation texture decreased. Increasing deformation presumably gives the wider distribution of cell sizes and orientations within the deformed grains which provides the cube-oriented nuclei. The higher stored energy provides an increased driving force for the growth of these cube-oriented nuclei, which may also have a special orientation relationship with the deformed structure which favours their growth relative to components of the deformation texture [8].

Stefán *et al.* [5], in studies of recrystallization in Fe–51 % Ni and Fe–75 % Ni alloys, observed that the primary recrystallization texture was the cube texture, and this became stronger in the course of normal grain growth prior to secondary recrystallization. In an earlier paper [9] they state that secondary recrystallization only occurs in matrices containing a strong cube-texture component. Their results are in general agreement with the present work, except for the presence of an appreciable proportion of retained deformation texture in the present work. This difference could be due to the fact that their material was cold-rolled to 99% reduction, compared with 60% in our case. Greater deformation favours the production of cube texture, as observed in the present work. Also, their material contained varying amounts of carbon and oxygen and had a small grain size, of about 15 μm, compared with those in the present work of 50 to 150 μm, depending upon annealing temperature. It has often been observed that increased impurity content favours the formation of cube texture in the primary recrystallization of Ni–Fe alloys [9].

Fig. 10 shows the influence of annealing temperature on the initial permeability, the annealing time being 4 h. Below 500° C there is a slight increase in permeability due to recovery, and between 500° C and approximately 900° C the permeability increases rapidly as the amount of recrystallization increases. After a 4 h anneal at 900 to 950° C complete primary

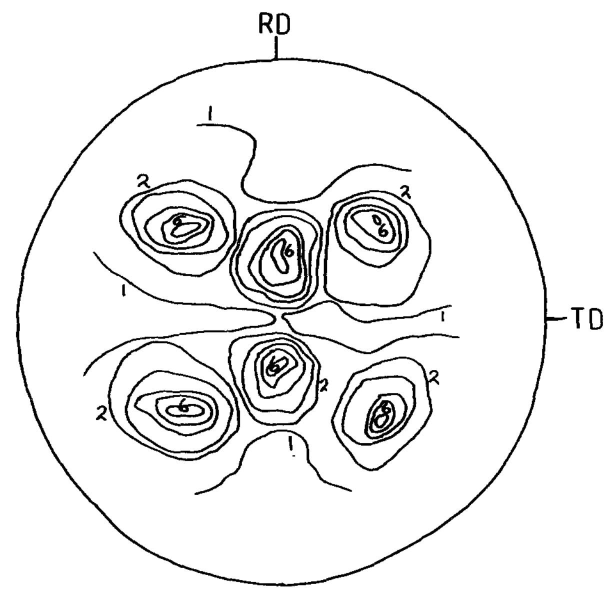

Figure 5 {1 1 1} pole figure for material annealed for 4 h at 1050° C.

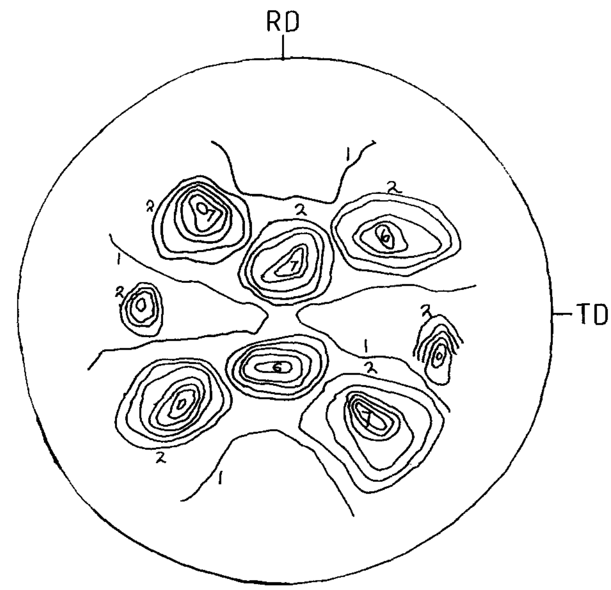

Figure 7 {1 1 1} pole figure for material annealed for 4 h at 1100° C.

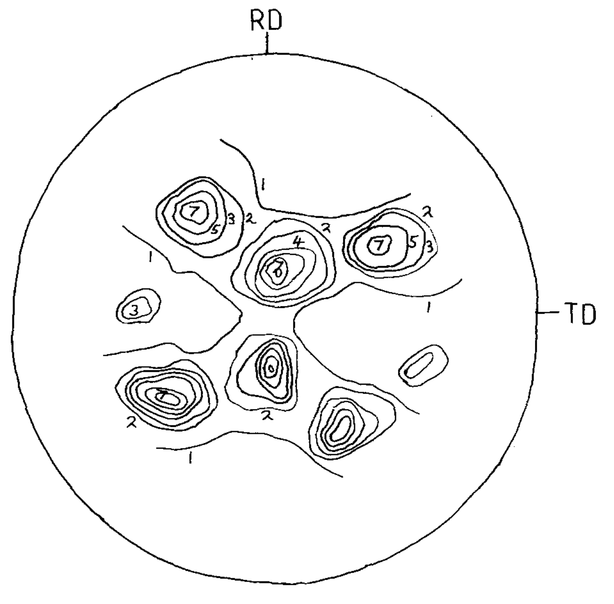

Figure 8 {1 1 1} pole figure for material annealed for 6 h at 1100° C.

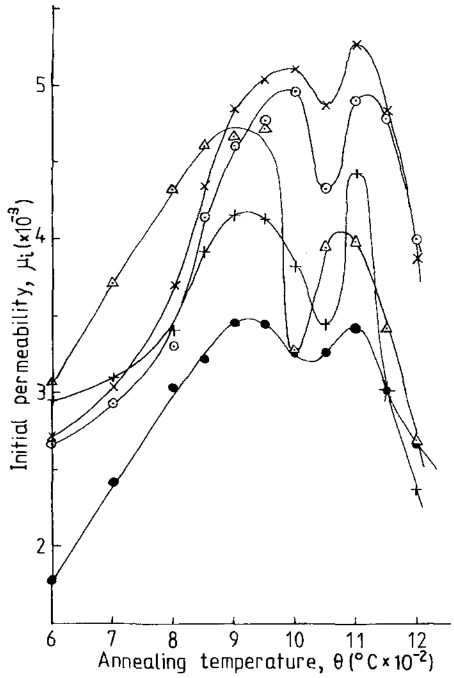

Figure 10 Variation of initial relative permeability with annealing temperature (4 h anneal). ⊙ Alloy A, (+) Alloy B, (△) Alloy C, (x) Alloy D, (●) Alloy E.

recrystallization has occurred, but only a weak texture has developed. Between 950 and 1050° C both the retained deformation texture and the cube texture develop, and the permeability decreases. The cube texture develops at the expense of the retained deformation texture between 1050 and 1100° C, and the permeability increases. Therefore the decrease in permeability between about 950 and 1050° C must be associated with the retained deformation texture, and the increase above 1050° C with the increased proportion of cube texture. The decrease in permeability is most likely to arise because in the retained deformation texture a significant proportion of the magnetization in the specimens cannot be close to the preferred "easy" crystallographic directions, which are ⟨1 0 0⟩ in this material, whereas the subsequent increase will arise because a larger proportion of the ⟨1 0 0⟩ easy directions of magnetization lie in the plane of the sheet when the cube texture has its maximum intensity, and the permeability again reaches a maximum value near this temperature. Above about 1100° C secondary recrystallization occurs, the cube texture is destroyed and the permeability decreases.

The variation of the saturation magnetostriction constant λ_s with annealing temperature is shown in Fig. 11 for material with 4.18 % Mo, the annealing time being 4 h. Annealing below 900° C produces little change in the measured magnetostriction constant, but there is a sharp decrease above this temperature to a minimum at 950° C, followed by a steady increase. The change in magnetostriction constant above 900° C must be associated with the formation, and relative proportions, of the two types of annealing texture discussed above. Below 900° C the amount of texture formed is negligible, which results in the value of the saturation magnetostriction being fairly constant, the approximately isotropic nature of the

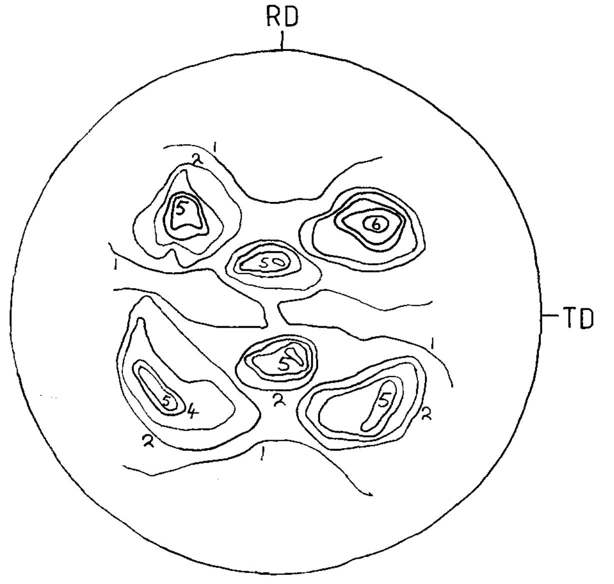

Figure 9 {1 1 1} pole figure for material annealed for 4 h at 1150° C.

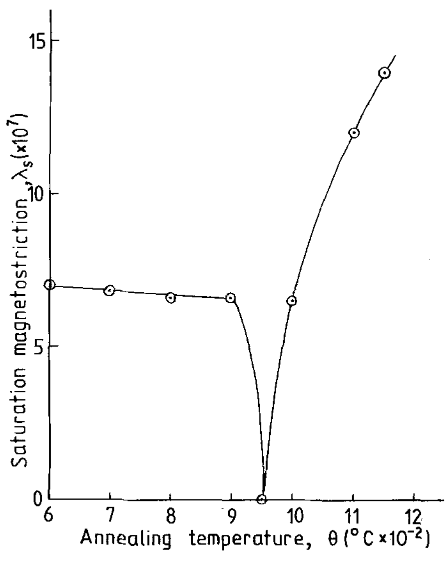

Figure 11 Variation of saturation magnetostriction constant with annealing temperature (4 h anneal).

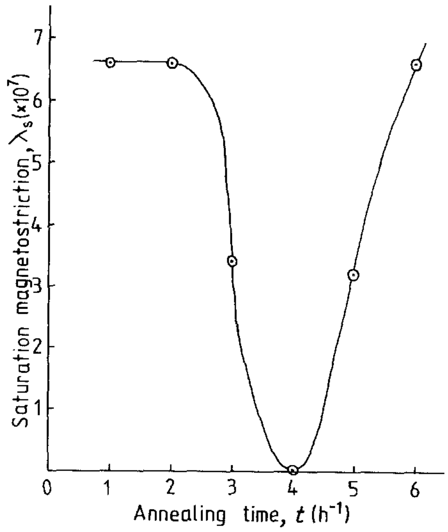

Figure 12 Variation of saturation magnetostriction constant with annealing time at 950° C.

material giving a value of magnetostriction constant averaged over all crystal orientations.

In binary Ni–Fe alloys near to Ni_3Fe, λ_{100} has about twice the value of λ_{111}. Although the magnitudes of λ_{100} and λ_{111} in quaternary alloys will differ from those in binary alloys, and will depend upon the amount of short-range order, λ_{100} would be expected to be greater than λ_{111}. The retained deformation texture will produce more directions in the plane of the sheet which lie close to $\langle 1\,1\,1 \rangle$ than to $\langle 1\,0\,0 \rangle$ and will therefore tend to give a lower mean value of magnetostriction constant than the cube texture, which produces a larger proportion of $\langle 1\,0\,0 \rangle$ directions in the plane of the sheet. Above about 900° C, λ_s begins to decrease and this must be associated with the development of the retained deformation texture. As the cube texture begins to predominate at higher temperatures, the magnetostriction reaches a minimum value and then increases. Above about 1100° C the cube texture is reduced by secondary recrystallization, but this does not appear to significantly reduce λ_s at 1150° C. Fig. 12 shows the variation of λ_s with annealing time at 950° C. The decrease in λ_s between 2 and 4 h annealing times may be explained, as above, by the stronger development initially of the retained deformation texture. The subsequent increase is the result of the cube texture becoming dominant at longer times.

A decrease in λ_s will tend to increase the permeability. Therefore the changes in permeability due to anisotropy and magnetostriction oppose one another for the two types of texture. The effect of anisotropy must, however, be dominant until the temperature is reached at which the texture is destroyed by secondary recrystallization. Above this temperature, about 1100° C, the steady increase in λ_s shown in Fig. 11 could account for the decrease in permeability shown in Fig. 10. At the highest annealing temperatures, oxidation due to an insufficiently dry hydrogen atmosphere could also contribute to the fall in permeability.

The permeability, magnetostriction and texture results are therefore consistent with one another.

The measurements of permeability and magnetostriction described above were made on specimens which were furnace-cooled after annealing. As the material cools through the temperature range from approximately 460 to 400° C, short-range order formation will occur and tend to reduce the anisotropy and magnetostriction [6]. It would therefore be expected that quenching of the specimens after annealing would increase the effects of texture on permeability and magnetostriction. Conversely, a furnace cool, or low-temperature anneal, to produce the optimum amount of short-range order which makes the anisotropy and magnetostriction practically zero would decrease the effect of texture.

Similar measurements on Fe–36 wt % Ni alloys, with additions of molybdenum, chromium and copper, have been reported earlier [4]. The results showed that the permeability reached a maximum at about 900 to 950° C, when the recrystallization texture was fully developed. However, the recrystallization texture showed no cube component, and the double maximum in permeability was not obtained because there were not the two competing texture components observed in the 77 % Ni alloys.

4. Conclusions

Annealing of some cold-worked permalloys of approximate wt % composition 77 Ni, 14 Fe, 5 Cu and 4 Mo, has been shown to have a marked effect on the initial permeability due to recrystallization, texture formation and secondary recrystallization.

The permeability increases rapidly with annealing temperature between 500 and 900° C due to primary recrystallization, the amount of texture formation being too small to significantly affect the permeability in this temperature range.

Above about 900° C, two annealing textures develop in competition with one another; a modified deformation texture which tends to decrease the permeability and a cube texture which increases the permeability. Up to about 1050° C the two textures develop approximately equally and the permeability decreases. Above 1050° C the cube texture predominates, giving an increase in permeability until the texture is destroyed by secondary recrystallization above about 1100° C and the permeability falls. The resultant effect of these processes is that a 4 h anneal produces maximum permeabilities at about 950 and 1100° C, with an intermediate minimum. The temperatures at which the maximum and minimum occur vary somewhat with composition.

Different proportions of easy $\langle 1\,0\,0 \rangle$ directions lie close to the plane of the sheet in the two types of texture which develop, and this is thought to be responsible for their opposite effects on the permeability.

Changes in magnetostriction constant can also be related to the texture in a similar way to changes in permeability.

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