JOURNAL OF MATERIALS SCIENCE 29 (1994) 742–748

Extrusion processing of FeCo

L. ZHAO, I. BAKER
Thayer School of Engineering, Dartmouth College, Hanover, NH 03755, USA

Ingots of three FeCo alloys, Fe–70 at % Co, Fe–50 at % Co and Fe–30 at % Co, were successfully extruded at 1000 °C, then re-extruded at 750 °C in order to produce fine-grained material. The resulting microstructures were examined both optically and by transmission electron microscopy. It was found that increasing the Fe:Co ratio produces less complete recrystallization: Fe–70 at % Co was completely recrystallized even after both the 1000 and 750 °C extrusions, whilst Fe–30 at % Co was partially recrystallized after the 1000 °C extrusion. Single dislocations were present in the extruded alloys except single-extruded Fe–30 at % Co, in which dislocation pairs were observed.

1. Introduction

FeCo, a B2-type intermetallic compound which undergoes an order–disorder transformation is the basis of a number of high-saturation, low-permeability magnetic alloys. The alloys ranging from 35–50 at % Co have the highest room-temperature saturation magnetization of any material [1]. However, the binary alloys, especially the stoichiometric alloy, are brittle in the ordered state [2–4]. Thus, hot-forging or hot-rolling are normally used to modify the microstructure of ingots [1, 2, 5]. Some investigators have even found that the alloy is difficult to hot forge [6, 7].

Recently, some intermetallic compounds, Ni_3Al, NiAl, FeAl and Fe_3Al, have been successfully hot-extruded to rods in either stainless steel or mild steel cans (see, for example, [8]). In order to study the effects of disordering and Fe : Co ratio on ductility and grain-size strengthening in FeCo, hot-extrusion of ingots was also used to produce a fine grain size in three alloys: Fe–70 at % Co, Fe–50 at % Co and Fe–30 at % Co. This paper presents the microstructures of these alloys after extrusion.

2. Experimental procedure

Ingots of Fe–70 at % Co, Fe–50 at % Co and Fe–30 at % Co, 50 mm diameter and 200 mm long, were cast at the United Technologies Research Center, East, Hartford, CT, after vacuum induction melting. The ingots were extruded twice at the NASA–Lewis Research Center: the first extrusion was at 1000 °C in stainless steel cans at an area reduction ratio 7.5:1, whilst the second extrusion was at 750 °C in 1030 mild steel cans at an area reduction ratio of 4:1. The phase diagram for Fe–Co indicates that all the alloys are f c c (αCo, γFe) at 1000 °C and b c c (αFe) at 750 °C. No cracks were observed in either the ingots or the extruded rods.

The microstructures of the extruded FeCo alloys were examined optically in both transverse and longitudinal sections. Specimens for optical microscopy were etched in a solution of 30% nitric acid in methanol. Microhardness tests were performed on unetched specimens using a Leitz Wetzlar microhardness tester with a 100 g load; each value given is the average of 20 tests.

TEM samples cut perpendicular to the extrusion direction were electro-polished in a solution of 10% perchloric acid in methanol at − 30 °C at 12 V with a medium flow rate. The samples were washed in methanol immediately after electro-polishing. All the samples were viewed in a Jeol 2000FX operating at 200 kV. A considerable problem with TEM examinations of the FeCo is its highly magnetic nature which often shifts the electron beam off the screen. It was often very difficult to return the beam to the screen. The only way to do this was to tilt the specimen. This problem made dislocation analysis in the TEM almost impossible.

3. Results

3.1. Fe–50 at% Co

The microstructure of single-extruded Fe–50 at % Co as observed optically is shown in Fig. 1. Equi-axed grains with a grain size of 50–60 μm were present in transverse sections, see Fig. 1a, whilst in longitudinal sections, grains were elongated along the extrusion direction, see Fig. 1b. The average thickness of these lenticular grains was about 80 μm. Dark spots were observed inside grains and along grain boundaries. However, there appears to be no particular correspondence of the spots with the grain boundaries. These dark spots were holes left by particles with a high concentration of silicon and small amount of aluminium during polishing [4]. Strong contrast was also observed between different grains, indicating the etching of this alloy depends strongly on the orientations of the grains.

The first extrusion produced a low residual dislocation density ($5 \times 10^{11} m^{-2}$) in Fe–50 at % Co, see Fig. 2. Most of the dislocations present were single dislocations. Because dislocations in the ordered state are $a/2\langle 1\,1\,1 \rangle$pairs [3], it is reasonable to assume that

0022–2461

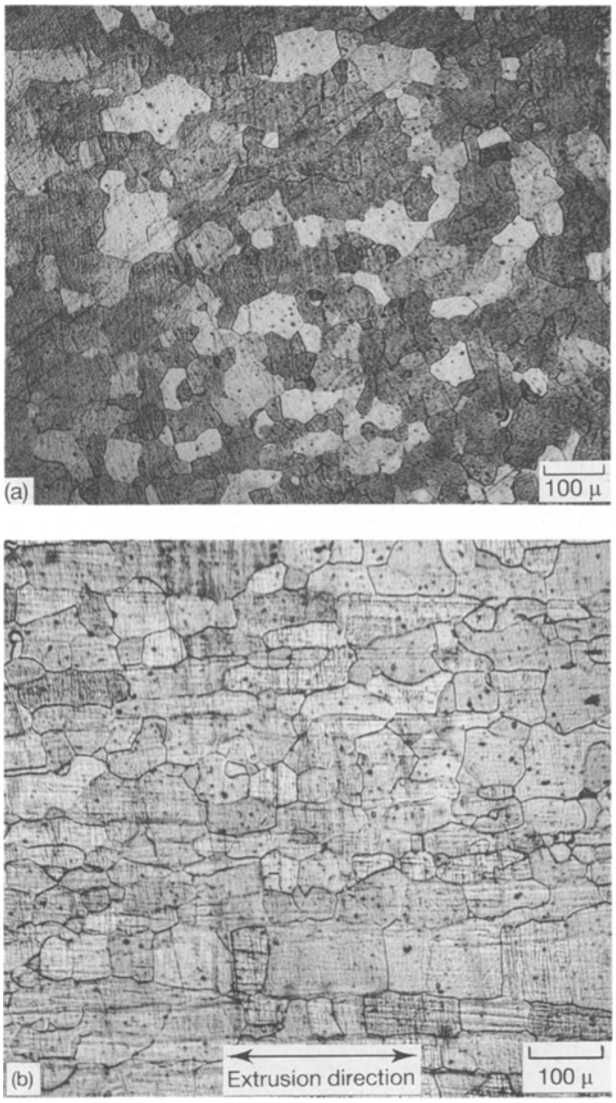

Figure 1. Optical micrographs of single-extruded Fe–50 at % Co. (a) Transverse section, (b) longitudinal section.

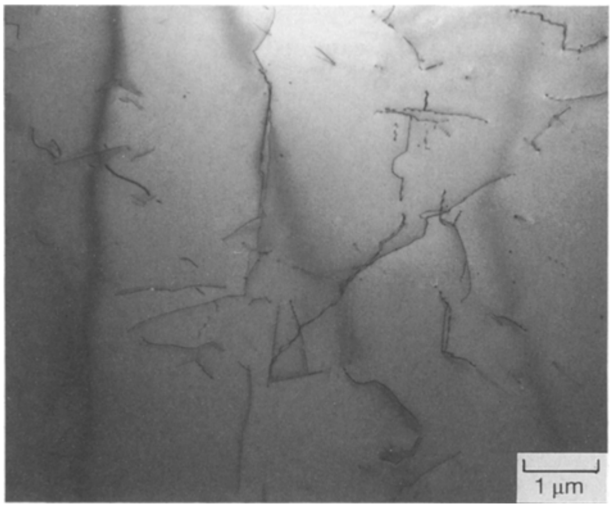

Figure 2. Transmission electron micrographs of single-extruded Fe–50 at % Co in transverse section, showing a low dislocation density.

the alloy was bcc before extrusion – the ingot was heated to 1000 °C but almost certainly cooled to below 985 °C, the fcc/bcc transformation temperature, before extrusion.

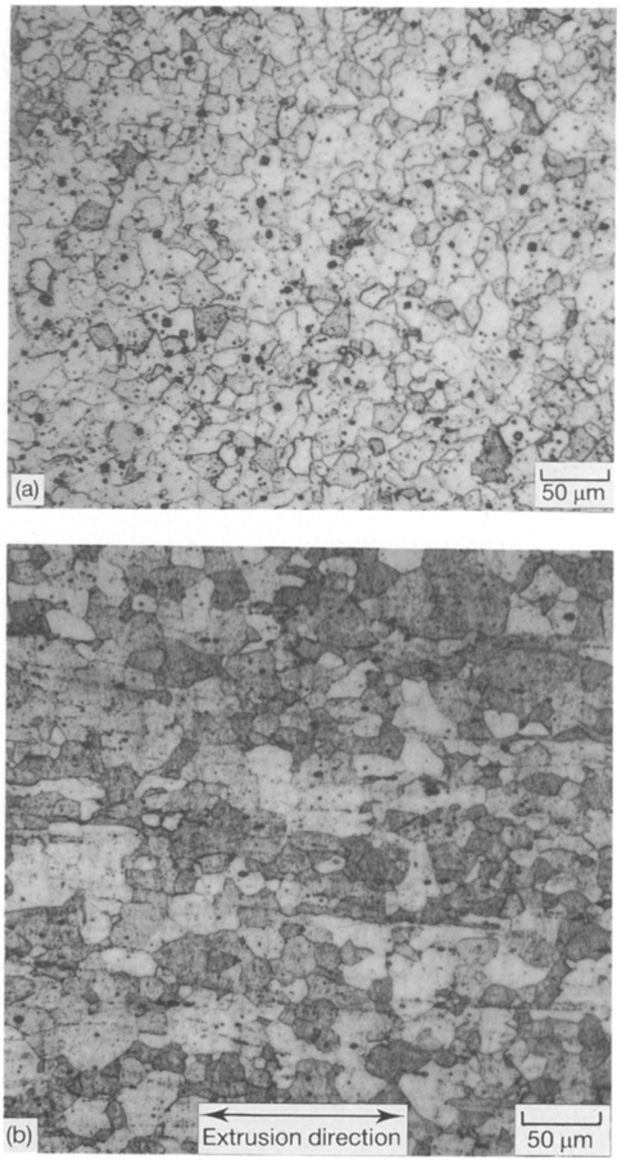

Figure 3. Optical micrographs of double-extruded Fe–50 at % Co. (a) Transverse section, (b) longitudinal section.

Fig. 3. shows the microstructure of the double-extruded Fe–50 at % Co. In transverse sections, grains as well as indistinct grain-boundary areas were present (Fig. 3a). The estimated grain size was about 10–20 µm. As shown in Fig. 3b, the microstructure in longitudinal sections was similar to that in transverse sections. However, compared with transverse sections, the microstructural scale was larger. Lines of particles in longitudinal sections were aligned along the extrusion direction.

TEM observations of the double-extruded Fe–50 at % Co showed groups of unrecrystallized grains and recrystallized grains of 1–2 µm diameter (Fig. 4a). These probably correspond to the indistinct grain-boundary areas in the optical micrographs. Some of the small grains showed fringe contrast at their boundaries, typical of high-angle boundaries, whilst others were subgrains and the boundaries consisted of dislocation networks. These observations indicate that the material was partially recrystallized and diffusion-assisted processes were operating during

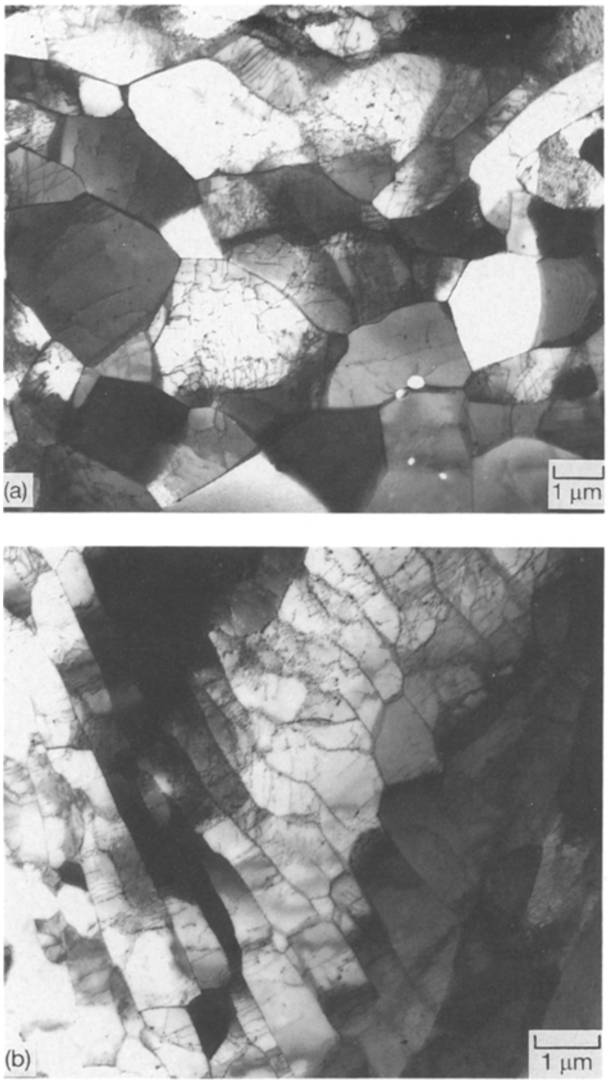

Figure 4. Transmission electron micrographs of double-extruded Fe–50 at % Co in transverse section, showing grain groups: (a) group of small equi-axed grains, and (b) groups of small elongated grains.

the extrusion. Fig. 4b shows elongated subgrains. These subgrains had a high dislocation density. That they are subgrains is evinced by the small change in contrast from one grain to the next and the dislocation walls separating the grains.

3.2. Fe–70 at% Co

Single-extruded Fe–70 at % Co showed an equi-axed grain structure in transverse sections (Fig. 5a). The grain size, at about 20–30 µm, was smaller than that of single-extruded Fe–50 at % Co. Dark spots were also observed inside grains as well as along some grain boundaries. Fig. 5b shows the microstucture of a longitudinal section. Only a few elongated grains were present. Again some black spots were aligned along the extrusion direction, although they did not lie on the grain boundaries. The grain size in longitudinal sections was 30–40 µm, which was about 10 µm larger than that in transverse sections. TEM observations again revealed a very low dislocation density, suggesting the material had a fully recrystallized structure.

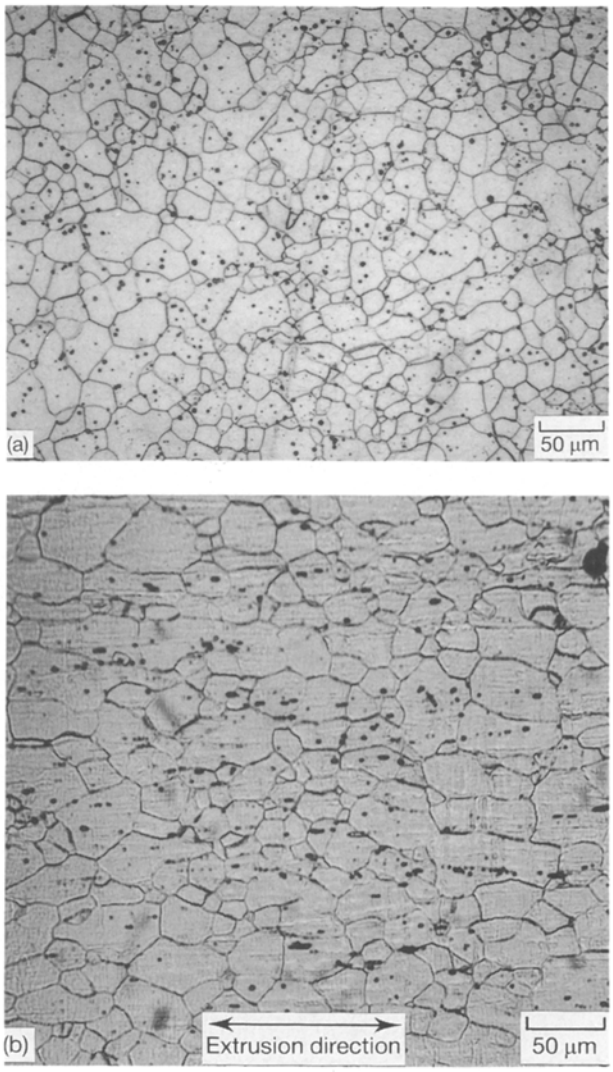

Figure 5. Optical micrographs of single-extruded Fe–70 at % Co. (a) Transverse section, (b) longitudinal section.

Fig. 6 shows optical micrographs of double-extruded Fe–70 at % Co. This microstructure is similar to that of single-extruded Fe–70 at % Co. However, in contrast to the first extrusion, the second extrusion produced a more uniform microstructure because (1) the grain size, 20–30 µm, in transverse sections was the same as in longitudinal sections; (2) no elongated grains were present in longitudinal sections and (3) although the alloy had almost the same grain size in transverse sections after each extrusion, grains upto 50 µm were present after the first extrusion while large grains after the second extrusion were about only 40 µm. TEM also revealed a very low dislocation density after the second extrusion, suggesting the material was fully recrystallized.

3.3. Fe–30 at% Co

Fig. 7 shows optical micrographs of single-extruded Fe–30 at % Co. Instead of appearing as equi-axed grains with clear boundaries, the grains were flattened in transverse sections (Fig. 7a). It was noted that grains closer to the centre of the extruded rod had smaller dimensions. A more uniform microstructure was present in longitudinal sections (Fig. 7b), and

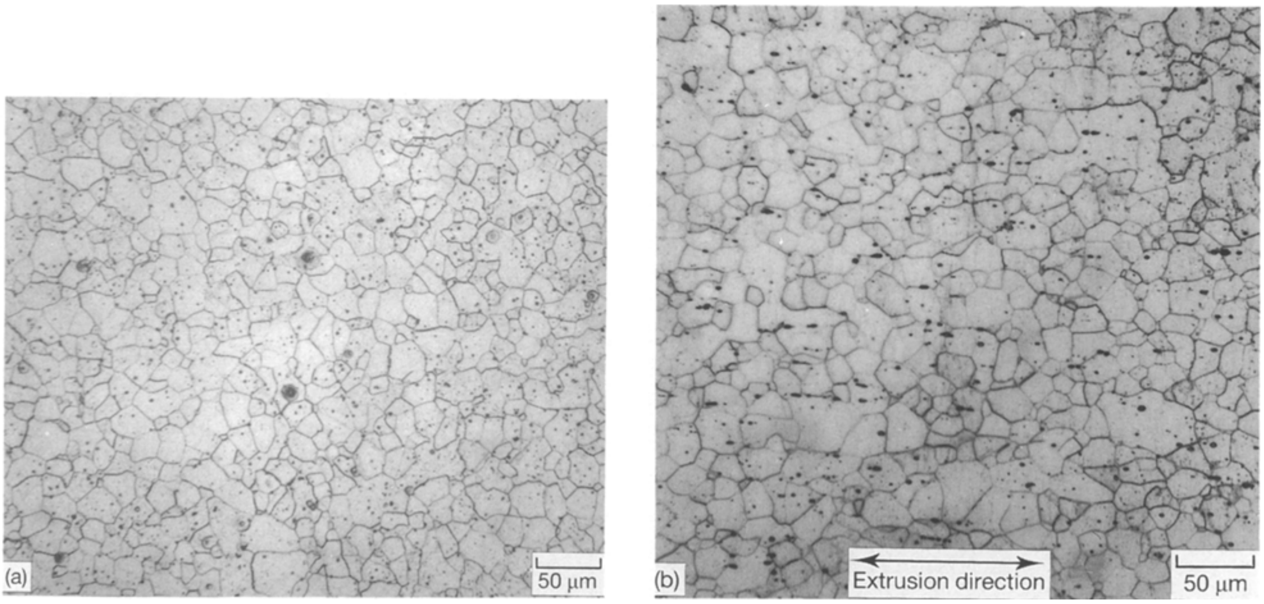

Figure 6. Optical micrographs of double-extruded Fe–70 at % Co. (a) Transverse sections, (b) longitudinal sections.

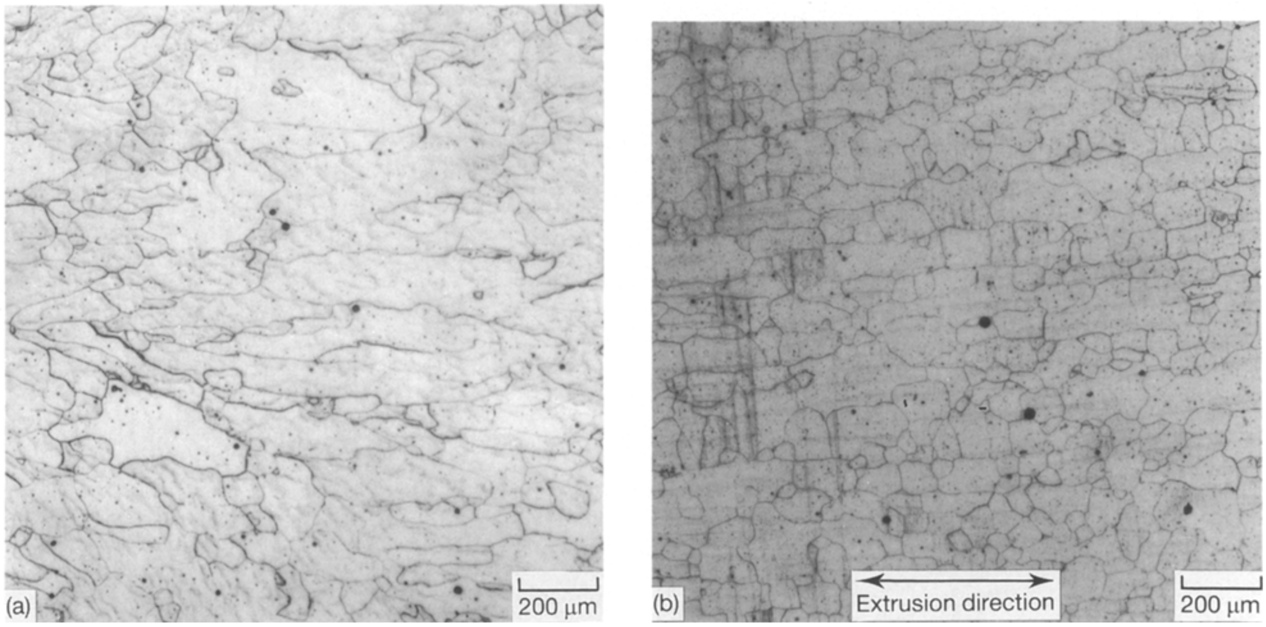

Figure 7. Optical micrographs of single-extruded Fe–30 at % Co. (a) Transverse section, (b) longitudinal section.

although most grains were equi-axed, a few large grains were elongated along the extrusion direction. The grain size obtained from longitudinal sections was about 100–110 μm. Small particles were also observed in this alloy. TEM observations showed that the first extrusion produced a higher dislocation density ($3 \times 10^{12}\,m^{-2}$) in the Fe–30 at % Co than in the other alloys. Most dislocations were in pairs, see Fig. 8a. Moreover, dislocation networks were observed (Fig. 8b), which formed low-angle boundaries, suggesting the material was partially recrystallized. Fig. 8c shows helical dislocations, caused by interactions between vacancies and dislocations. Both the helical dislocations, and dislocation networks are indicative of diffusion and dislocation climb.

Fig. 9 shows optical micrographs of double-extruded Fe–30 at % Co. One characteristic of transverse sections was the presence of individual grains which etched well along with more indistinct areas (Fig. 9a). The microstructure was similar to that of double-extruded Fe–50 at % Co. Optical micrographs of longitudinal sections, see Fig. 9b, were similar to those in transverse sections. The difference was that some of the indistinct areas in longitudinal sections were elongated along the extrusion direction. The grain size after the second extrusion was estimated to be about 20–40 μm, i.e. much smaller than that after the first extrusion. TEM also revealed the presence of groups of unrecrystallized grains and very fine grains (Fig. 10a) and dislocation networks (Fig. 10b) in

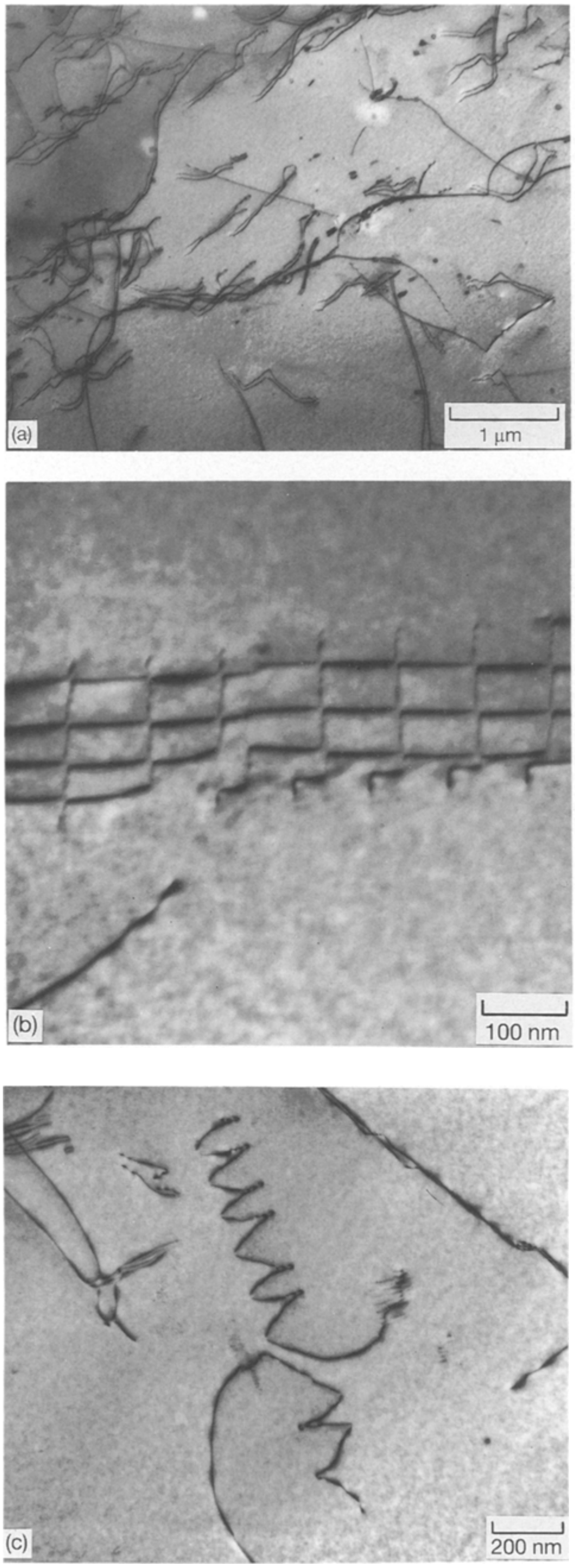

Figure 8. Transmission electron micrographs of single-extruded Fe–30 at % Co in transverse sections, showing a high dislocation density. (a) Dislocation pairs, (b) dislocation network, (c) helical dislocations.

double-extruded Fe–30 at % Co. Single dislocations were observed.

Table I shows the Vickers microhardness and a summary of the grain sizes of the extruded FeCo

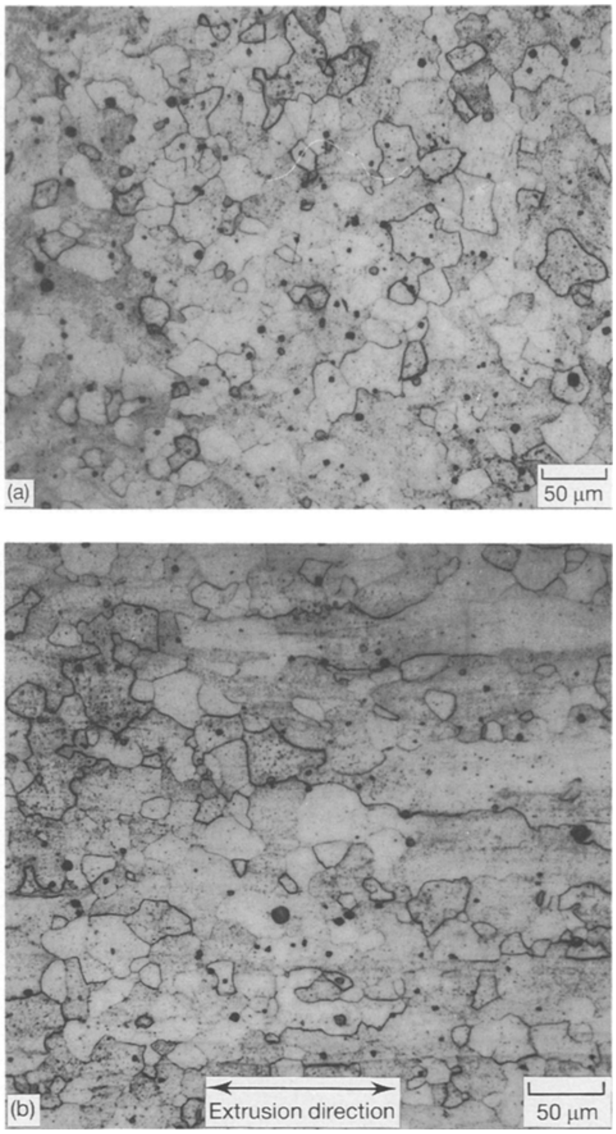

Figure 9. Optical micrographs of double-extruded Fe–30 at % Co. (a) Transverse section, (b) longitudinal section.

alloys. The stoichiometric alloy has a higher microhardness than the off-stoichiometric alloys. An interesting feature is that there was no significant difference in the microhardness of each alloy after the two extrusions even though the grain sizes are quite different.

4. Discussion

Observations of the microstructures of the three FeCo alloys showed that the cobalt-rich alloy, Fe–70 at % Co, was fully recrystallized after both the first and second extrusions, the stoichiometric alloy Fe–50 at % Co was fully recrystallized only after the first extrusion but not after the second extrusion, whilst the iron-rich alloy Fe–30 at % Co was partially recrystallized even after the first extrusion. This indicates that the cobalt-rich alloy, Fe–70 at % Co, dynamically recrystallized more easily than the other two alloys. One question is: does the recrystallization of the FeCo alloys depend on their phase transformation temperatures or melting temperatures?

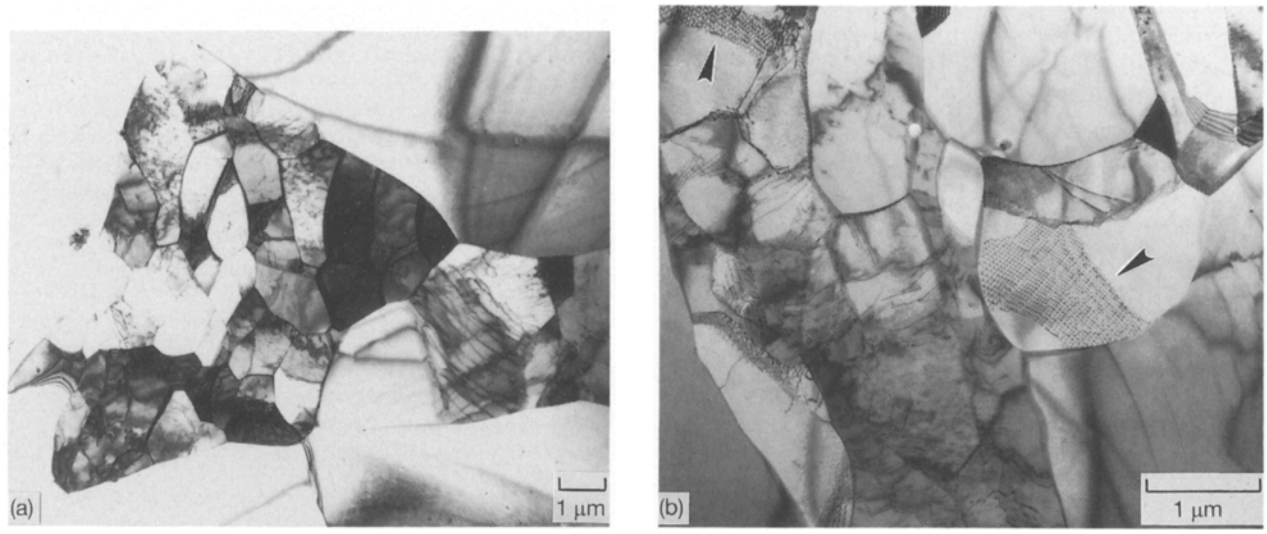

Figure 10. Transmission electron micrographs of double-extruded Fe–30 at % Co in transverse sections, showing (a) a group of small equiaxed grains, and (b) dislocation networks (arrowed).

TABLE I Microhardness and grain size of extruded FeCo

	Single-extruded Fe–70 at % Co	double-extruded Fe–70 at % Co	single-extruded Fe–50 at % Co	double-extruded Fe–50 at % Co	single-extruded Fe–30 at % Co	double-extruded Fe–30 at % Co
Microhardness, H_v	238	243	266	269	217	221
Grain size (µm)	20–40	20–30	50–60	10–20	100–110	20–40

According to the Fe–Co phase diagram [9], the stoichiometric alloy has a higher order–disorder and fcc–bcc transformation temperatures than the two off-stoichiometric alloys and the melting point is almost the same for the three alloys. This suggests that the recrystallization of FeCo alloys does not show a trend with the order–disorder transformation temperature, fcc–bcc transformation temperature or melting point. A priori one might have expected stoichiometric FeCo to be the most difficult to recrystallize because diffusion is probably slowest in this alloy.

As each composition was extruded under the same extrusion conditions, i,e. the same extrusion temperature and the same amount of deformation, the difference in recrystallization among the three compositions reflected the difference in their properties. The driving force for recrystallization is the stored energy produced by deformation. Some of the stored energy will be released by recovery which occurs before recrystallization. It has been widely accepted that recovery is a polygonization process and low-angle grain boundaries are produced. The evidence of low-angle grain boundaries in both single-extruded and double-extruded Fe–30 at % Co indicates that this alloy recovered easily, thereby reducing the driving force for recrystallization.

In FeCo alloys, the stoichiometric alloy has the highest order–disorder transformation temperature 730 °C, which is, however, considerably lower than the first extrusion temperature 1000 °C. Therefore, it is surprising to observe dislocation pairs in the single-extruded Fe–30 at % Co.

5. Conclusion

All the three FeCo alloys, Fe–70 at % Co, Fe–50 at % Co and Fe–30 at % Co, could be successfully hot-extruded to rods in cans at both 750 °C and 1000 °C. The first extrusion at 1000 °C produces a smaller grain size, about 30µm, in the cobalt-rich alloy than in the stoichiometric alloy and the iron-rich alloy which have a grain size of 50–60, and 100µm, respectively. The second extrusion at 750 °C produces a grain size of about 20–40 µm in all three alloys. Increasing the Fe : Co ratio leads to less complete recrystallization: the cobalt-rich alloy is completely recrystallized after both the 1000 and 750 °C extrusions, whilst the iron-rich alloy, Fe–30 at % Co, is partially recrystallized even after the 1000 °C extrusion. Single dislocations are present in the extruded alloys except single-extruded Fe–30 at % Co, in which dislocation pairs are observed.

Acknowledgements

The authors thank Dr D. L. Anton, United Technologies Research Center for providing the ingots, and Dr J. D. Whittenberger NASA–Lewis Research

Center, for extruding them. The use of the Electron Microscope Center at Dartmouth College is also acknowledged. This work was supported by the US Department of Energy through Grant DE-FG02-87ER45311.

References

1. R. V. MAJOR and V. SAMADIAN *J. Mater. Eng.* **11** (1989) 27.
2. M. J. MARCINKOWSKI and H.CHESSIN, *Philos. Mag.* **10** (1964) 837.
3. A. M. GLEZER and I. V. MALEYEVA, *Phys. Met. Metall.* **68** (1989) 65.
4. LIE ZHAO, M.S. THESIS, Thayer School of Engineering, Dartmouth College (1992).
5. D.W. CLEGG and R.A. BUCKLEY, *Met. Sci. J.* **7** (1983) 48.
6. J.K. STANLEY, *Trans. ASM* **42** (1950) 150.
7. K. KAWAHARA, *J. Mater. Sci.* **18** (1983) 1709.
8. I. BAKER and P.NAGPAL, in "Processing and Fabrication of Advanced Materials for High Temperature Application-II", edited by T.S. Srivatsen and V.A. Ravi, (The Metallurgical Society, Warrendale, Pa., 1993) p. 3.
9. T. MASSALSKI (ed), "Binary Alloy Phase Diagram", (American Society for Metals, Materials Park, Ohio, 1986).

Received 29 September 1992
and accepted 8 June 1993