Effect of thermomechanical treatment on the properties of Fe-11Al and Fe-14Al alloys

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Fe-Al alloys have the potential to be relatively inexpensive soft magnetic materials if their formability could be improved. An investigation has been made on the effect of thermomechanical treatment on the properties of Fe-11 wt%Al and Fe-14 wt%Al alloys (designated Fe-11Al and Fe-14Al respectively). For the former the room temperature mechanical properties were found to be determined principally by the recrystallised grain size. A good combination of properties for Fe-11Al, i.e. high strength and ductility, was obtained when the grain size was less than about 100 μm. The small grain size was produced by warm rolling at 600°C followed by 1 hour annealing at 600–700°C. On the other hand hot rolling followed by annealing resulted in large grain size, hence rendered the alloy brittle. The cold formability also exhibited a grain size dependence, with the Fe-11Al alloy with a fine recrystallised grain size having good cold rollability. In contrast Fe-14Al was brittle irrespective of the treatment given; ductility of less than 1% was observed in all cases and the cold rollability was limited. Ordering was not seen to be a factor affecting the observed mechanical properties and rollability of either alloy as all the thermomechanical treatments, other than an ordering treatment of 500 hours at 400°C, resulted in a disordered structure. The stress required to work these alloys at elevated temperatures were estimated from compression tests and it is apparent that for Fe-11Al the stress is greatly reduced (50%) from the room temperature value at 600°C and that at 750°C both alloys required a similar stress which was about 15% of the room temperature value. The magnetic properties of Fe-11Al compared favourably with Fe-14Al; the former has a higher saturation induction, a similar coercive force but a lower permeability than Fe-14Al.

1. Introduction

Iron aluminium alloys are of interest as low cost, soft magnetic materials. However, as yet, they have not been commercially exploited due the difficulty in fabrication into the thin sheet that is required for most soft magnetic applications. Many studies have been conducted to improve formability [1–9] but despite some progress, the problem remains incompletely resolved; the alloys can be hot worked but show limited ductility at room temperature [5–9]. Most of these studies were conducted on or around Fe_3Al intermetallic composition and the poor formability and ductility have been commonly associated with ordering that takes place within this compositional range. The poor room temperature ductility has also been attributed to the result of environmental embrittlement in the presence of water vapour [10, 11].

In contrast, research on alloys with lower aluminium content, i.e. disordered α-phase alloys, has been limited. Alloys of this composition offer no less attractive properties than the intermetallic composition both in mechanical and magnetic properties. In fact as far as the mechanical properties are concerned, they are likely to offer a solution to the poor room temperature ductility encountered in the intermetallic composition while maintaining a reasonably high strength. The magnetic properties of the α-phase offers a compromise between the decreasing saturation induction and the increasing permeability and resistivity with increasing aluminium content [12].

This paper reports the development of such an alloy focusing on the effect of thermomechanical treatments on its properties. For comparison purposes, an alloy of Fe_3Al composition was also studied.

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TABLE I Chemical analysis (wt%) of Fe-11Al and Fe-14Al alloys

Alloy	Fe	Al	C	Si	S	P	Mn	Ca	O_2	N_2
Fe-11Al	Bal.	10.450	0.020	<0.010	<0.001	0.001	<0.010	<0.001	0.006	0.004
Fe-14Al	Bal.	13.910	0.015	<0.010	<0.001	0.002	<0.010	<0.001	0.011	0.007

2. Experimental procedure

2.1. Alloy preparation

Alloys of Fe-11 wt%Al and Fe-14 wt%Al (i.e., Ni_3Al composition) nominal compositions, hereafter called Fe-11Al and Fe-14Al respectively, were produced from spectrographically pure elements. The raw materials, 60 grams in total weight for each alloy, were melted in an arc melter under a reduced-pressure argon atmosphere. To reduce oxygen contamination, a titanium ingot was used as a gas getter; the ingot was melted before and after melting the alloy constituents. The alloys were melted three times to ensure homogeneity and then moved into a finger-shaped mould where they were remelted twice to produce a smooth, finger-shaped ingot of about 100 mm long and 100 mm^2 in cross-section. The compositions of the alloys are given in Table I and can be seen to be close to the nominal compositions.

2.2. Hot and warm rolling

Prior to hot rolling, the ingot was machined to remove surface oxides and protrusions and to give parallel faces; the ends of the ingot were also discarded. The slab produced was then heated for 2 hours at 950°C and rolled on a laboratory two-high mill. The mill was set to give 1 mm thickness reduction per pass. Ten to fifteen minutes were normally allowed for reheating to 950°C between the passes. The procedure was repeated to obtain a sheet of about 2.5 mm thick (75% reduction). A part of the sheet was sometimes further rolled to 1 mm (90% reduction). Warm rolling was carried out at 600°C. Except for the temperature, the procedure was the same as that for the hot rolling.

2.3. Heat treatment and cold rolling

Subsequent to hot/warm rolling, heat treatments of 1 hour duration were carried out at temperatures in the range 600–1000°C inclusive under a dynamic argon atmosphere. Prior to cold rolling, the heat-treated sheets were cleaned by lightly grinding their surfaces to remove the oxide layer. Cold rolling was carried out to various thickness reductions to determine the material's cold formability.

2.4. Mechanical properties

Flat tensile specimens having gauge dimensions of 13 mm × 5.5 mm were stamped from hot- and warm-rolled strips of 1 mm thick. The tensile axis was made parallel to the rolling direction. The specimens were annealed for 1 hour at various temperatures between 600–1000°C under a static argon atmosphere. Tensile tests were conducted at room temperature at a cross-head speed of 0.78 mm/min which corresponds to a strain rate of approximately 1×10^{-4} s^{-1}.

Isothermal, constant strain rate compression tests were performed on square cross-section specimens having nominal dimensions of 2.5 mm × 2.5 mm × 5 mm. The specimens were machined from warm rolled alloys with the compression axis parallel to the rolling direction. The specimens were annealed at 700°C for 1 hour to recrystallise with small grain size. The testing was carried out at a strain rate of 0.01 s^{-1} and over a range of temperature between room temperature to 750°C. Samples were loaded cold and then heated at a rate of 25°C min^{-1} to the test temperature. To reduce the friction at the specimen loading face, the specimens were lubricated with boron nitride.

2.5. Structure

The microstructure of the thermomechanically treated samples was examined using light and scanning electron microscopy. For light microscopy, the samples were polished to a 1 μm diamond finish and etched in a 20 vol% HNO_3 + 80 vol% HCl solution to reveal the grain structure. The grain size was determined by the linear intercept method. One hundred grains or more, depending on the grain size, were counted on each sample. The scanning electron microscope was also used to examine the fracture surface of tensile specimens.

2θ-scans using Cu K_α radiation were carried out on a Philips diffractometer (PW1710) to determine whether ordering occurred after heat treatment. The degree of order was determined by measuring the long-range order parameter, S, which for Fe_3Al ordered structure is given by

$$S = \sqrt{\frac{I_{(200)}/I_{(400)}(\text{Meas.})}{I_{(200)}/I_{(400)}(\text{Cal.})}}$$

where $I_{(200)}$ and $I_{(400)}$ are the intensities of superlattice and fundamental peaks respectively. The numerator refers to the measured intensities, whereas the denominator refers to intensities obtained from fully ordered alloy.

The texture was measured using a Philips X'pert system with Cu K_α radiation. The orientation distribution function $f(g)$ was computed from three measured pole figures, namely (110), (200) and (211) pole figures.

2.6. Magnetic measurement

AC magnetic measurements were made on heat-treated ring samples. The samples were punched from 50% cold rolled sheets of around 0.6 mm thick and annealed at 1050°C for 4 hours in a dry hydrogen atmosphere. The measurements were carried out at a field frequency of 50 Hz.

TABLE II Effect of thermomechanical treatment on the cold rollability of Fe-11Al and Fe-14Al alloys

Alloy	Thermomechanical treatment	Grain size (μm)	Cold rollability
Fe-11Al	Warm rolled 75%	Deformed structure	Did not roll
	Warm rolled 75% + annealed at 700°C for 1 hr	30	Rolled without cracking to >90% deformation
	Warm rolled 75% + annealed at 900°C for 1 hr	220	Rolled up to 80% deformation
	Warm rolled 75% + annealed at 1000°C for 1 hr	380	Did not roll
	Hot rolled 75% + annealed at 1000°C for 1 hr	210	Cracked after about 20% deformation
	Hot rolled 90% + annealed at 700°C for 1 hr	150	Rolled to 50% deformation without cracking.
Fe-14Al	Warm rolled 90% + annealed 700°C for 1 hr	70	Rolled up to about 50% deformation
	Hot rolled 90% + annealed at 700°C for 1 hr	165	Failed at early stage

3. Results

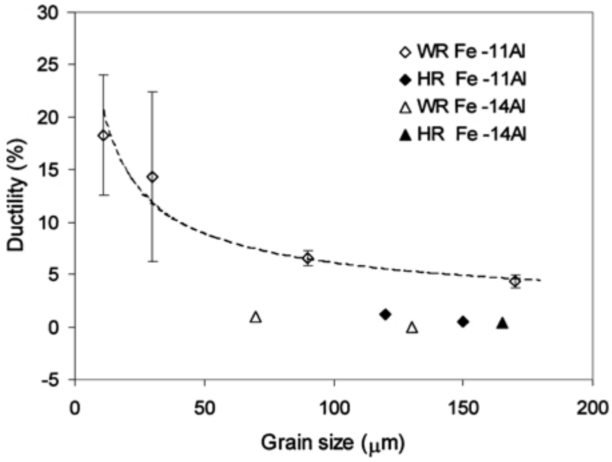
3.1. Rolling

Both alloys could be hot or warm rolled; no cracks were observed in any of the samples rolled to 90% reduction. However, not all of the samples could be easily cold rolled. Hot rolled and heat treated samples of either alloy were generally very difficult to cold roll and failed by extensive cracking at 50% reduction or less (Table II). In contrast, warm rolled specimens of Fe-11Al could generally be cold rolled if they were given a suitable annealing treatment following warm rolling. Annealing at 700°C for 1 hr was found to be the most suitable heat treatment to obtain good cold rollability. This schedule also appeared to be suitable for Fe-14Al although cracking still occurred at 50% reduction. Also presented in Table II is the grain size of the samples; there is an indication of a grain size dependence of the cold rollability for Fe-11Al and perhaps for Fe-14Al. However, for a given grain size, hot rolled and annealed samples seemed to be more brittle than the warm rolled and annealed samples.

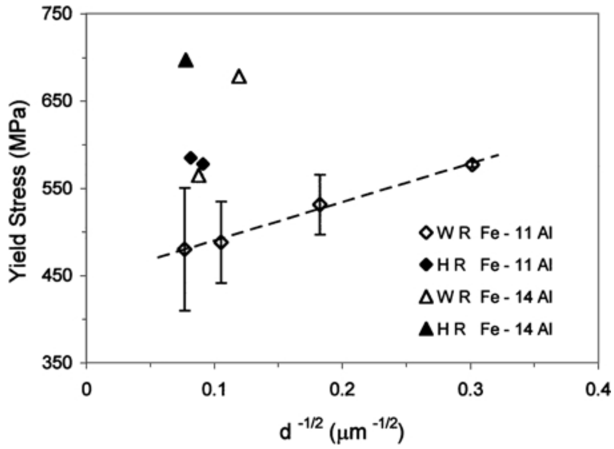
3.2. Mechanical properties

The room temperature tensile properties, together with the recrystallised grain size, of Fe-11Al and Fe-14Al alloys after a variety of thermomechanical treatments are given in Table III. The tensile properties of heat treated warm rolled Fe-11Al are mean values of three tests, whereas the data for the other conditions including those for Fe-14Al were obtained from one test. It is evident that the recystallised grain size is strongly determined by the annealing temperature; the higher the annealing temperature the larger the grain size. The data also demonstrated that, for a given annealing temperature, hot rolled materials had a larger grain size than that of the corresponding warm rolled materials. The variation in tensile properties with the annealing temperature for Fe-11Al can be attributed to a grain size effect as shown by the graphs of Fig. 1. Due to the limited amount of data for Fe-14Al, it is not possible to comment on whether a similar grain size dependence exists.

As shown in Fig. 1a, the warm rolled and annealed Fe-11Al exhibited a very strong grain size dependence of ductility; the ductility fell sharply with the increase in grain size (up to ~100 μm), but the rate of decrease became less significant at larger grain sizes. It is interesting to note that even with a large grain size (>100 μm), warm rolled and annealed Fe-11Al was still reasonably



(a)



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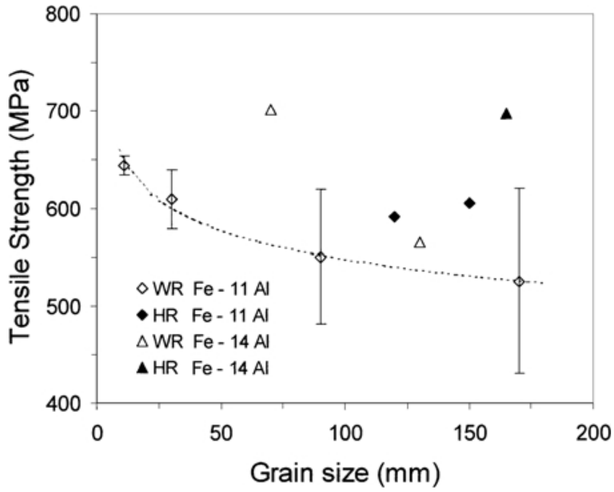


(c)

Figure 1 Effect of grain size on the tensile properties of Fe-11Al and Fe-14Al alloys. WR = warmed rolled, HR = hot rolled.

ductile with a ductility of around 5%. In contrast, in the hot rolled and annealed condition, the alloy was very brittle, i.e. the ductility was less than 1% (albeit only two data points were available).

The Fe-14Al alloy exhibited practically no ductility; both specimens, one warm rolled and annealed and the other hot rolled and annealed, exhibited a ductility of less than 1%.

The yield stress values of warm rolled and annealed Fe-11Al were lower than those after hot rolling and annealing treatments. Furthermore, the Fe-11Al had a lower yield stress than the Fe-14Al. For Fe-11Al there is a significant grain size effect on the yield stress that was consistent with the Hall-Petch [13, 14] relationship (Fig. 1b). For both alloys the tensile strength followed a similar pattern to the yield strength, i.e. warm rolling and annealing gave lower strength values than those obtained after hot rolling and annealing (Fig. 1c). Fig. 1c also demonstrates that the tensile strength of Fe-11Al is grain size dependent, but that the dependency follows a similar trend to that exhibited by ductility rather than yield stress.

The scanning electron microscopy study of the fracture surfaces from the room temperature tensile test specimens of Fe-11Al and Fe-14Al revealed that both alloys fractured predominately in a transgranular mode (cleavage fracture) irrespective of their ductility, which ranged from a fraction of a percent to around 20% (Fig. 2). However, the cleavage features differed; brittle samples exhibited large cleavage planes (corresponding to their large grain size) with a marked river pattern (Fig. 2b and c), whereas those that were ductile (i.e. Fe-11Al warm rolled 90% and annealed at 600–700°C for 1 hour) showed irregular, smaller cleavage planes (corresponding to their smaller grain size) with some isolated voids and dimpled regions (Fig. 2a).

Fig. 3 shows the stress-strain curves as a function of temperature for Fe-11Al and (also the curve at 750°C for Fe-14Al). In all tests the onset of plastic deformation was followed by a slowly decreasing stress with increasing strain (e.g. the 500°C curve) or an almost constant stress (e.g. 750°C curve). The maximum flow stress as a function of temperature is presented in Fig. 4; the most noticeable feature being the rapid fall in stress at temperature in excess of 400°C. At 750°C the flow stress of Fe-11Al and Fe-14Al was similar in contrast to about 20% difference at room temperature as indicated by the tensile test data (see Fig. 1 and Table III).

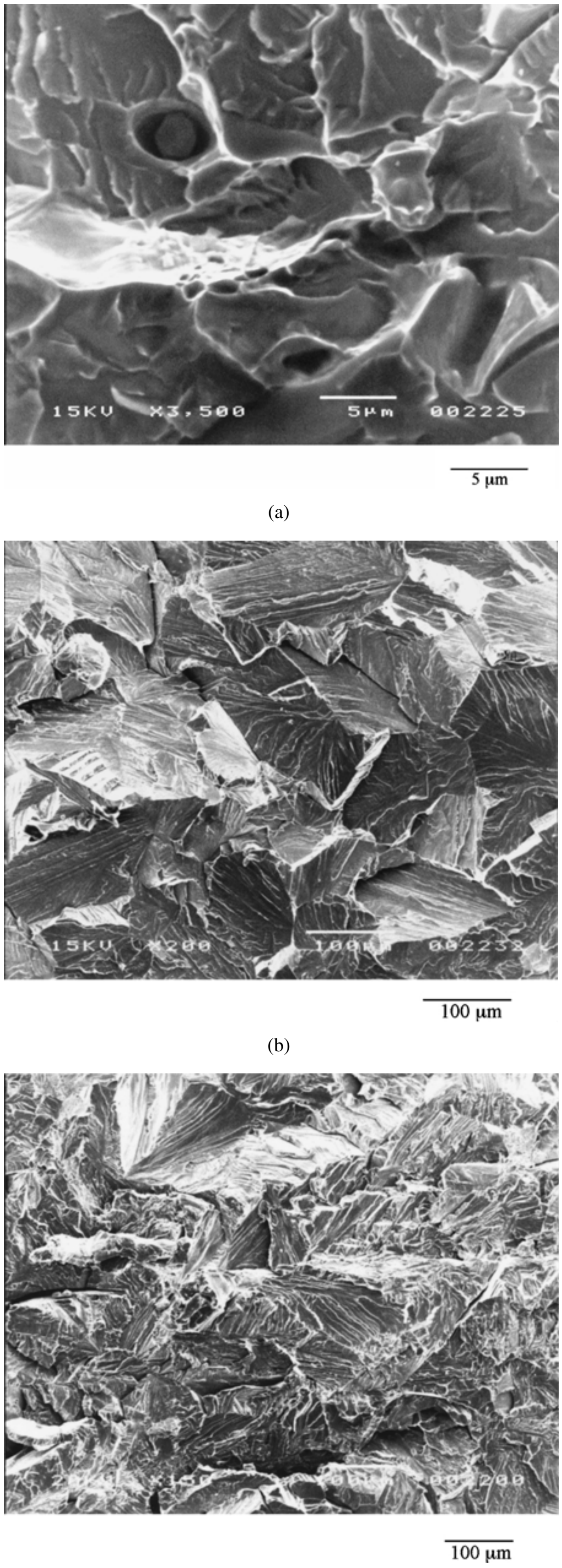


Figure 2 Scanning electron images of fracture surfaces from room temperature tensile tests: (a) Fe-11Al warm rolled 90% and annealed at 600°C for 1hr showing cleavage fracture with some void formation. The material had a small grain size (11 μm) and fractured at 20% ductility, (b) Fe-11Al hot rolled 90% and annealed at 700°C for 1hr showing cleavage fracture. The material had a large grain size (150 μm) and fractured at 1.2% ductility and (c) Fe-14Al warm rolled 90% and annealed at 700° for 1hr showing cleavage fracture. The material had a large grain size (165 μm) and fractured at 0.4% ductility.

3.3. Structure

Hot rolling resulted in an equiaxed grain structure. In contrast the as-warm rolled material had a deformed structure, but annealing for 1 hour at temperatures of 600°C and above produced a recrystallised microstructure.

X-ray diffraction demonstrated that neither alloy subjected to the thermomechanical treatments described in Section 2.4 prior to mechanical testing exhibited ordering. A typical diffraction pattern is inset in Fig. 5a, and shows no sign of the presence of superlattice peaks. Therefore in order to compare the ordering tendencies

TABLE III Room temperature tensile properties of Fe-11Al and Fe-14Al alloys

Alloy	Thermomechanical treatment	Grain size (μm)	Tensile strength (TS) (MPa)	Yield stress (MPa)	Ductility (%)
Fe-11Al	WR 90% + 600°C 1 hr	11	644	577	18.3
	WR 90% + 700°C 1 hr	30	609	531	14.3
	WR 90% + 800°C 1 hr	90	550	488	6.5
	WR 90% + 900°C 1 hr	170	525	480	4.3
	HR 90% + 600°C 1 hr	120	591	578	0.5
	HR 90% + 700°C 1 hr	150	605	585	1.2
Fe-14Al	WR 90% + 700°C 1 hr	70	701	678	1
	WR 90% + 800°C 1 hr	130	565	~TS	~0
	HR 90% + 700°C 1 hr	165	697	~TS	0.4

WR = warm rolled, HR = hot rolled.

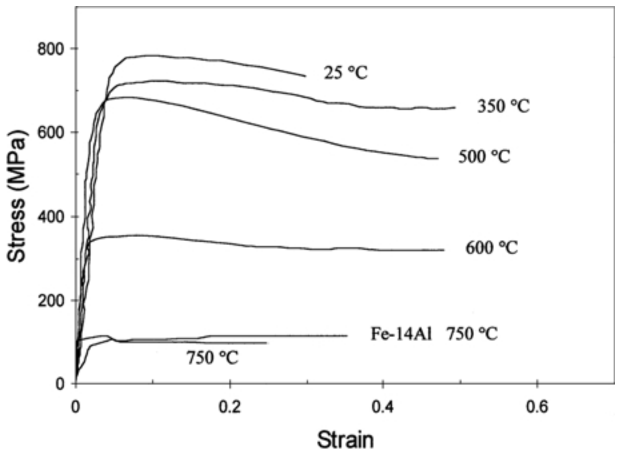


Figure 3 Compressive stress-strain curves at different temperatures.

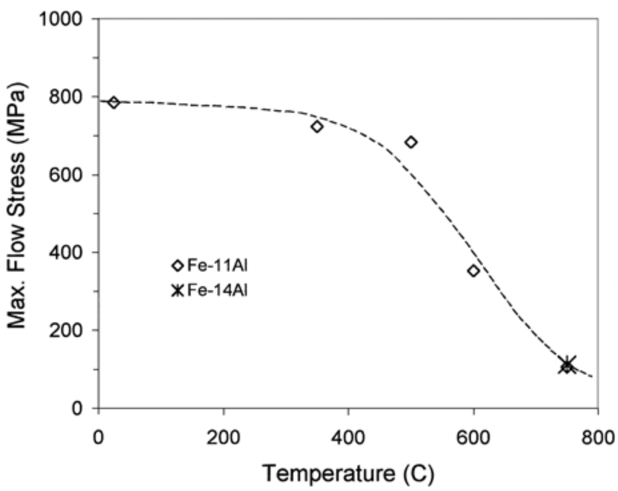


Figure 4 Temperature dependence of the maximum compressive flow stress.

of the two alloys an ordering heat treatment of 500 hours at 400°C was given. Fig. 5 shows the diffraction patterns for the alloys after the ordering heat treatment; a fully ordered structure ($S \approx 1$) was obtained for Fe-14Al but only a partial ordering ($S \approx 0.2$) for Fe-11Al.

Heat treated hot rolled and heat treated warm rolled Fe-11Al samples exhibited similar texture. However the texture was more marked in the latter. In both cases, the predominant texture components were (001)⟨110⟩, (111)⟨112⟩, (111)⟨110⟩ and (001)⟨100⟩ with the first component being much stronger than the rest (Fig. 6).

TABLE IV AC magnetic properties of Fe-11Al and Fe-14Al measured at 50 Hz. μ_{max} is the maximum permeability, H_c the coercivity, B_r the remanence, and B_s the saturation induction

Alloy	Grain size (μm)	μ_{max}/1000	H_c (Am^{-1})	B_r (T)	B_s (T)
Fe-11Al	330	1.88	235	0.33	1.52
Fe-14Al	310	3.37	218	0.47	1.22
Fe-11Al[a]	–	1.77	64	0.27	1.27
Fe-14Al[a]	–	3.59	57	0.36	1.16

[a]Data taken from reference [15], DC measurement.

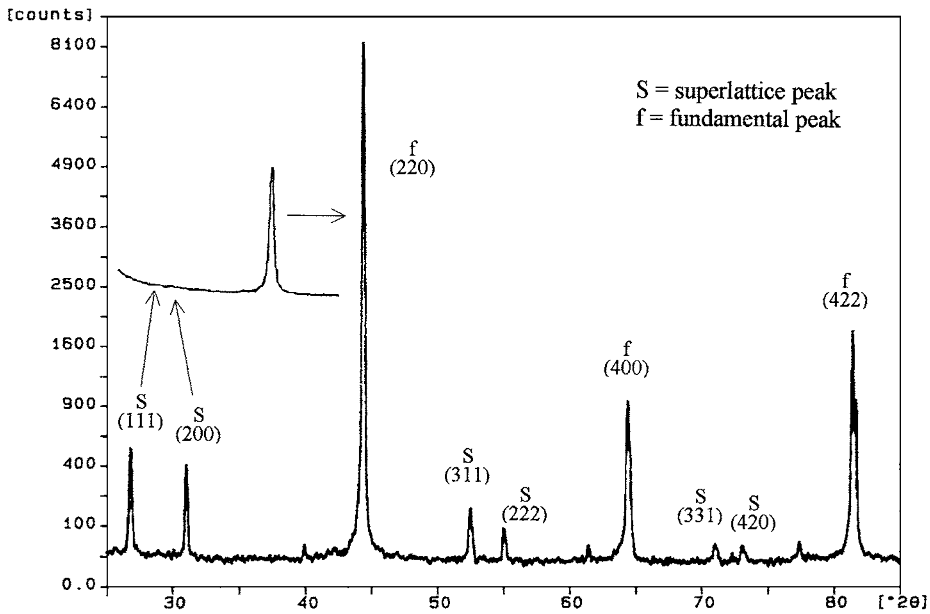
3.4. Magnetic properties

Fig. 7 and Table IV give the AC magnetic properties of the Fe-11Al and Fe-14Al alloys. Also presented in Table IV is the grain size of the samples used for the magnetic measurement; the grain sizes were similar. The two alloys exhibited striking differences in the maximum permeability (μ_{max}), the saturation induction (B_s) and the remanence (B_r) but their coercivity (H_c) values were about the same. The results from the work of Masumoto and Saito [15] for similar compositions and heat treatment (in the annealed state) are also given in Table IV.

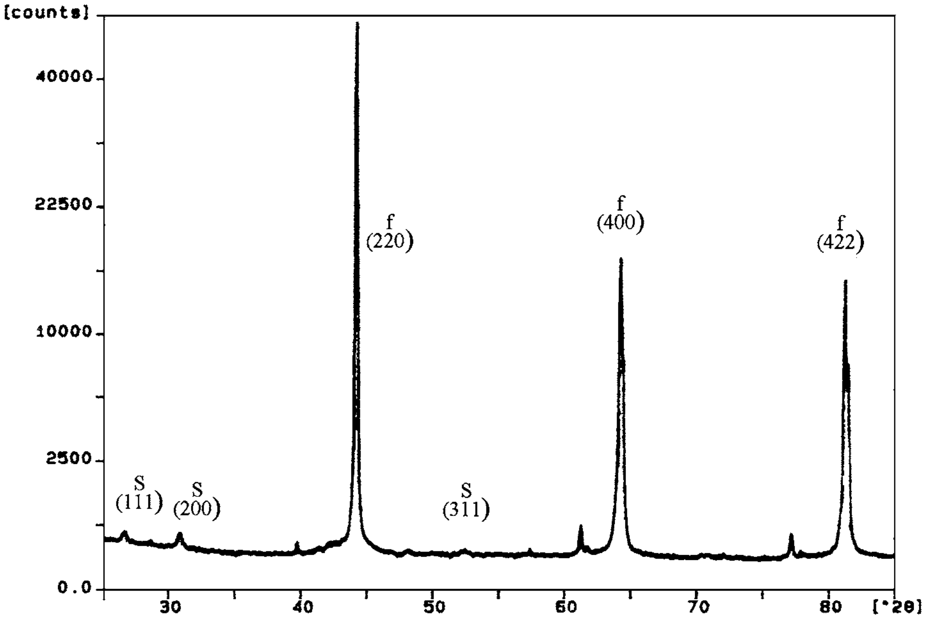
4. Discussion

Both alloys were successfully hot rolled at 950°C and warm rolled at 600°C, the former leading to equiaxed grains and the latter to a deformed grain structure. The rollability is associated with the marked decrease in flow stress that occurs at temperatures in excess of 400°C (Fig. 4). In the present investigation the temperature dependence of the flow stress was only fully studied for Fe-Al but Sharma *et al.* [16] found a similar softening trend in Fe_3Al based alloys, in which the hot hardness fell abruptly between 450 and 700°C and levelled off at a hardness value nearly a decade lower than the value at room temperature from 700°C onward.

It is important for industry to know the forces required for different rolling operations. The compression data indicate that the forces required for warm rolling at 600°C are approximately 50% of that for cold rolling. 600°C is the lowest feasible temperature for warm rolling as a few trials at lower temperatures of 400 and 550°C (not mentioned in the experimental procedure and result section) were unsatisfactory; the materials cracked after a few rolling passes. The lower limit



(a)



(b)

Figure 5 Effect of heat treatment on the ordering of Fe-14Al and Fe-11Al: (a) A fully ordered Fe_3Al structure in Fe-14Al obtained after annealing at 400°C for 500 hours. The inset is a part of a typical diffraction pattern obtained from a sample that had received a thermomechanical treatment (in this case 1000°C) prior to mechanical testing and shows a disordered structure. (b) A partial ordered ($S = 0.2$) in Fe-11Al obtained after annealing at 400°C for 500 hours.

of warm rolling temperature is in agreement with that found by Nachman and Buehler [1]. These workers suggested a temperature of 575°C to be the optimum condition for warm rolling of an Fe-16Al alloy. They assumed that ordering was responsible for the failure at lower temperatures (575°C is just above the order-disorder transition temperature [17]). However, ordering does not play a role in the present investigation on Fe-11Al as a long time at a low temperature was required to obtain a significant amount of long range order.

Compression tests were only performed up to 750°C but it is clear from our results and those of Sharma *et al.* [16] that the flow stress is levelling out at these temperatures. It is therefore possible to state that the forces for hot rolling at 950°C are about 15% of those required for cold rolling. In fact the recrystallisation observed on heat treatment at temperatures as low as 600°C and the levelling out of the flow stress at temperatures in excess of 750°C suggest that hot rolling may be performed at lower temperatures than that used in the present work. Indeed the actual hot working temperature in the present work was probably lower than 950°C as the rolls were not preheated and the workpiece was much smaller than the rolls.

Given a suitable heat treatment, warm rolled Fe-11Al could be cold rolled. Warm rolled Fe-11Al had a deformed grain structure and heat treatment caused recrystallisation. The rollability and ductility were found to be a function of the recrystallised grain size (Table II and Fig. 1); the smaller the grain size the greater the

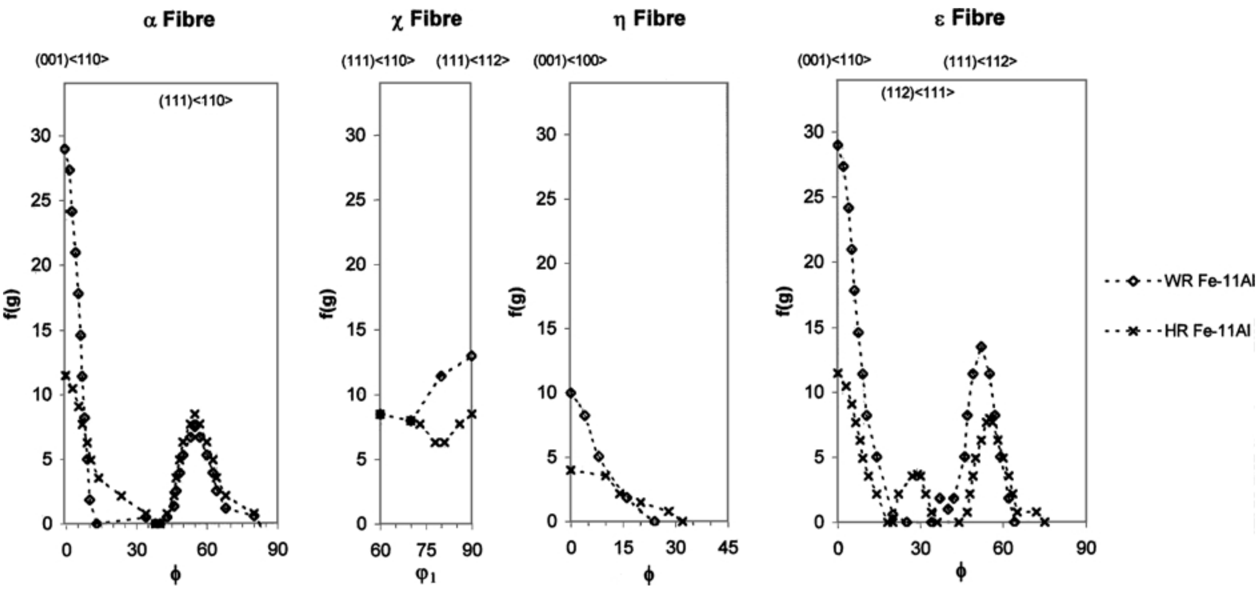


Figure 6 Texture components of heat treated warm rolled (WR) and heat treated hot rolled (HR) Fe-11Al.

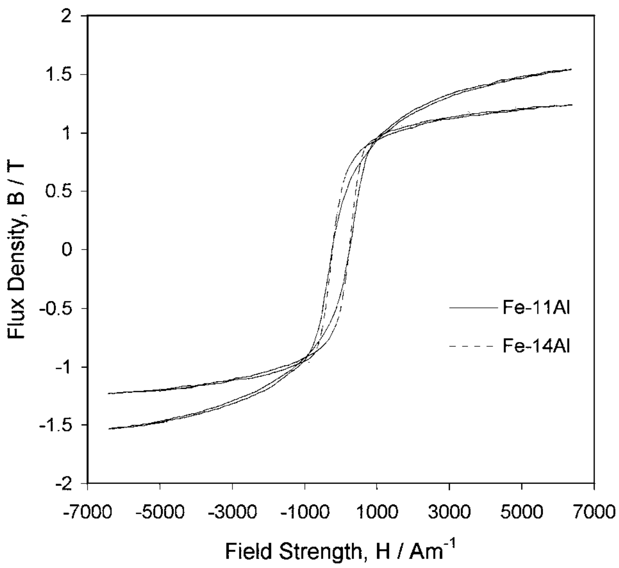


Figure 7 B-H loops at 50 Hz of Fe-11Al and Fe-14Al.

ductility and the more amenable was the material to cold rolling. Heat treated, warm rolled Fe-14Al was more brittle than the corresponding Fe-11Al material and did not cold roll so successfully. Ordering is commonly considered to be a major factor affecting the ductility of iron aluminium alloys of high aluminium content such as Fe-14Al [1, 4–9]. However, x-ray diffraction showed that the long-range order parameter was zero for heat treated, warm rolled Fe-14Al, thus long-range order cannot account for the brittleness of Fe-14Al in the present study. On the other hand, in agreement with previous workers [4, 18] who reported that the strength of Fe-Al alloys increased almost linearly with aluminium content up to 16%Al, present results showed that Fe-14Al had significantly higher yield and flow stresses than Fe-11Al. The high strength of Fe-14Al is attributed to solution hardening and perhaps also short-range order, which would not have been detected by the x-ray diffraction method employed. The high yield and flow stresses enhance brittleness.

Heat treated, hot rolled Fe-11Al specimens were more brittle than the corresponding warm rolled and heat treated materials of the same grain size (Fig. 1a). The only structural difference detected that may account for this behaviour is texture. The predominant texture in the heat treated hot rolled and heat treated warm rolled materials was the same but the latter showed a much stronger (001)⟨110⟩ component (Fig. 6). As shown in Fig. 2, the alloy had a cleavage mode of fracture. This type of fracture is controlled by the tensile stresses acting normal to a crystallographic cleavage plane, i.e. the resolved normal stress on the cleavage plane (σ_n). Having a bcc structure, the cleavage plane would be (100) planes. As mentioned in Section 2.4, the applied tensile stress was parallel to the rolling direction. It can be shown crystallographically that ⟨110⟩ rolling direction gives low values of σ_n, whereas most other directions give high values. Hence in a more random orientation (weak texture) as for the heat treated hot rolled material, the average value of (σ_n) would be higher than those for the heat treated warm rolled material. This may explain the difference in their ductility.

Changing the aluminium content results in a trade-off in the magnetic properties (Table IV). A decrease from 14% (Fe-14Al) to 11% (Fe-11Al) caused a decrease in permeability from 3.37 to 1.88 in agreement with the trend reported by Masumoto and Saito [15], and this is considered detrimental for a soft magnetic material. However, the same decrease in aluminium content has the beneficial effect of increasing saturation induction from 1.22 T to 1.52 T. A considerable decrease was also observed in the remanence on reducing the aluminium content, which also agrees with the Masumoto and Saito's results. The coercive force did not differ significantly but is about four times as high as Masumoto and Saito's results. The large discrepancy can be ascribed to the frequency effect; Masumoto and Saito used a DC method, whereas the present investigation employed an AC method at 50 Hz. In term of soft magnetic properties alone, the choice of alloy is very much determined by the specific application, i.e. depending on which property is paramount for the required performance. However for ease of fabrication Fe-11Al is clearly preferable.

5. Conclusions

The effect of thermomechanical treatment on the mechanical properties of Fe-11A1 and Fe-14Al has been studied. Fe-llAl exhibited a desirable combination of high strength and good ductility at room temperature (and hence good cold rollability) when treated to give a recrystallised grain size of $<100\ \mu$m. This has been produced by warm rolling at 600°C followed by annealing for 1 hour at 600–700°C. It was estimated that the forces required for warm rolling were about 50% of those required for cold rolling.

Hot rolling tended to produce a large grain size and hence rendered the alloy brittle. Furthermore for a given grain size the heat treated hot rolled materials were more brittle than heat treated warm rolled. The results indicated that hot rolling could perhaps be carried out at a lower temperature than that used (950°C) in the present work.

Fe-14Al was brittle in all cases, but exhibited higher strength than Fe-11Al. Long-range ordering was not seen to be responsible for the brittleness as all thermomechanical treatments (with the exception of a special ordering treatment of 500 hours at 400°C) yielded a disordered structure.

There is a trade-off in magnetic properties when changing from one composition to another but, in view of the ease of fabrication, Fe-11Al seems to be more attractive proposition than Fe-14Al.

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