Magnetic properties and thermomagnetic analysis of a Fe-20Mo-5Ni-0.075C alloy

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A Fe-20Mo-5Ni-0.075C (wt%) alloy for magnets was prepared by induction melting under vacuum. The material was hot rolled, solution treated and aged at 610 °C for different periods of time. The magnetic properties (H_c , B_r , B_s and (BH)_{max}) were measured and compared with some commercial alloys containing cobalt. The precipitation of Mo-rich phases and the decrease of the ferrite lattice parameter during ageing were detected by X-ray diffraction. The thermomagnetic analysis (TMA) was carried out in the solution treated samples and aged samples. The behaviour of TMA curves was explained with the help of X-ray analysis. © *1999 Kluwer Academic Publishers*

1. Introduction

Fe-Mo-Ni alloys have been reported as possible alternatives for the hard and semi-hard magnetic alloys containing cobalt (Vicalloy, Remalloy,...) [1]. One of the more interesting compositions is the Fe-20Mo-5Ni (wt %). When hot rolled, solution treated and quenched in oil or water, this alloy becomes very ductile [1, 2]. After solution treating the alloy must be aged at 550– 650 °C to achieve the best magnetic properties.

The precipitation reactions during ageing cause magnetic and mechanical hardening, which means, the coercive force (H_c) and the hardness are enhanced.

Magnetic and mechanical hardening were also observed in many Fe-Mo alloys [3–6]. According to Ustinovshikov *et al.* [4], the precipitation of a Laves phase λ (Fe₂Mo) was responsible for the mechanical hardening in Fe-10at.%Mo, Fe-15at.%Mo, Fe-20at.%Mo and Fe-26at.%Mo. The λ phase was also observed by Wan *et al.* [5] and Myiazaki *et al.* [6] in Fe-Mo alloys. According to Wan *et al.* [5] the magnetic hardening can be attributed to Mo-rich zones and/or fine λ particles.

Nickel is added to Fe-Mo alloys to provide high ductility to the material. On the other hand, it was observed [7] that the addition of 0.12 wt % C increased the coercive force while decreasing the ductility. Thus, a new composition, with a low carbon content, basically Fe-20Mo-5Ni-0.075C (wt %), was developed

with the expectation of improving both coercive force and ductility.

The magnetic properties of this alloy solution treated and aged were measured and compared to typical properties of some comercial alloys. X-ray diffraction and thermomagnetic analysis (TMA) were used to investigate the precipitations and to determine the transition temperatures in the material.

2. Materials and methods

An ingot with composition Fe-20Mo-5Ni-0.075C (wt %) was vaccum melted using high purity materials. The ingot was hot rolled (above $1000 \,^{\circ}$ C) with 90% of reduction divided in two steps. After rolling, the alloy was solution treated at $1230 \,^{\circ}$ C and oil quenched. Then, the samples were aged at $610 \,^{\circ}$ C for different times, up to ten hours.

The magnetic measurements (hysteresis loops in applied fields up to 10 kOe and thermomagnetic analysis up to $800 \,^{\circ}$ C) were carried out in a Vibrating Sample Magnetometer (VSM) EGG PAR model 4500. A barium ferrite was taken as standard for magnetic measurements.

The samples for magnetic measurements were disc shaped with diameter (c) of about 3.5 mm and thickness (a) between 0.05 and 0.20 mm. In the VSM the external magnetic field is parallel to the plane of the disc.

The hysteresis curves obtained were corrected for the demagnetization fields as proposed by Chikazumi [8] and Culity [9]. The values of B_r and $(BH)_{máx}$ become independent of the thickness of the samples only for the extremely fine ones.

TABLE I Coercive force $(_{\rm M}H_{\rm c})$, remanent and saturation inductions $(B_{\rm r} \text{ and } B_{\rm s})$ and energy product $(BH)_{\rm máx}$ of the Fe-20Mo-5Ni-0.075C

Condition	_M H _c (Oe)	<i>B</i> _r (kG)	B _s (kG)	(BH) _{máx} (MG Oe)	$B_{\rm r}/B_{\rm s}$
Hot rolled and solution treated	275	14.4	16.8	2.0	0.85
Aged at 610 °C/15 min	325	11.2	14.3	1.6	0.78
Aged at 610 °C/1 h	412	10.1	14.1	2.0	0.71
Aged at 610 °C/4 h	429	9.6	14.2	1.7	0.67
Aged at 610 °C/10 h	409	—	12.9	_	—

The X-ray diffraction data were carried out in a Philips diffractometer, using CuK_{α} radiation. The ferrite parameters were calculated using the {110} diffraction. The angles in the diffraction patterns have been measured step by step and the precision of the parameter calculations is higher than that required for 3 numerals.

3. Results and discussion

3.1. Magnetic properties

Table I presents coercivity ($_{\rm M}H_{\rm c}$), remanent and saturation inductions ($B_{\rm r}$ and $B_{\rm s}$, respectively) and energy product (BH)_{máx} of the material under the various conditions investigated.

As can be seen, as H_c increases B_r and B_s decreases with the ageing time. A decrease in B_s value with the ageing time was also observed by Wan *et al.* [5] in a Fe-16at.% Mo and by Jin and Tiefel [1] in the Fe-20Mo-5Ni (wt%) without carbon.

TABLE II Comparison of magnetic properties of Fe-20Mo-5Ni-0.075C and some commercial magnetic alloys containing Co [11]

Material	Magnetic properties				
	H _c (Oe)	$B_{\rm r}$ (kG)	B _s (kG)	(BH) _{máx}	
Fe-20Mo-5Ni-0.075C solution treated	275	14.4	16.9	2.0	
Fe-20Mo-5Ni-0.075C solution treated and aged 610 °C/1 h	412	10.1	14.1	2.0	
Steel 35%Co-3.75%W-5.75%Cr [11]	230	10.4	_	0.9	
Remalloy 2 12%Co-17%Mo-71%Fe [11]	340	8.6	—	1.2	
Vicalloy 1 10%V-52%Co-30%Fe [11]	240	8.6		0.9	
Vicalloy 2 13%V-52%Co-35%Fe [11]	415	9.1	_	2.3	

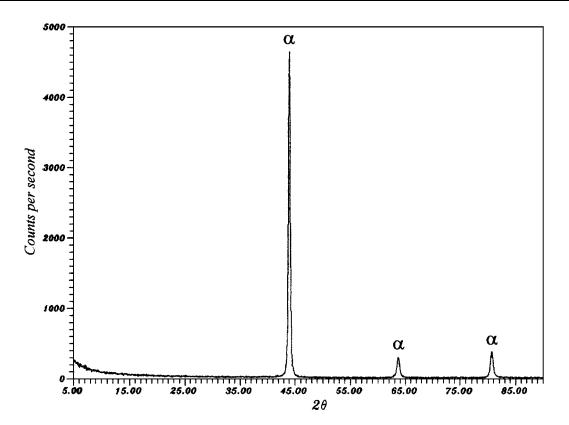


Figure 1 X-ray diffraction pattern of Fe-20Mo-5Ni-0.075C sample under the solution treated condition.

TABLE III Ferrite parameter as function of ageing time at $610\,^\circ C$

Condition	Solution treated	610°C/1 h	610°C/4h	610°C/10h
α lattice parameter (Å)	2.918	2.888	2.883	2.880

More details about the effects of the precipitation ageing on the magnetic properties of this alloy can be found in [10].

The best magnetic properties of the Fe-20Mo-5Ni-0.75C alloy were obtained after ageing for 1 h. However, the solution treated condition is also attractive

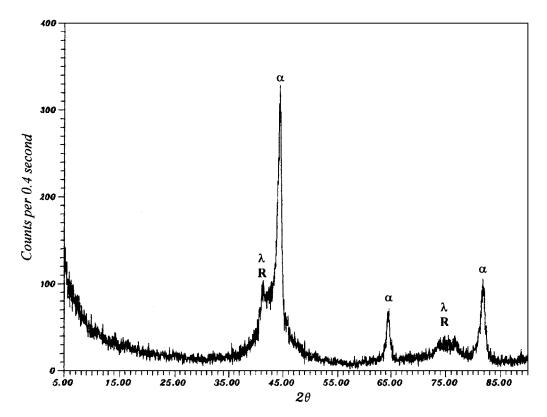


Figure 2 X-ray diffraction pattern of Fe-20Mo-5Ni-0.075C sample aged at 610 °C for 1 h.

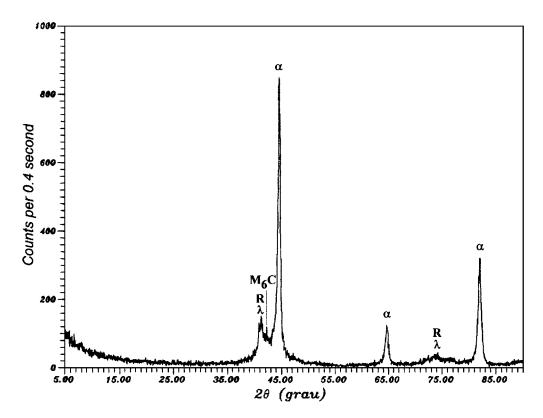


Figure 3 X-ray diffraction pattern of Fe-20Mo-5Ni-0.075C sample aged at 610 °C for 10 h.

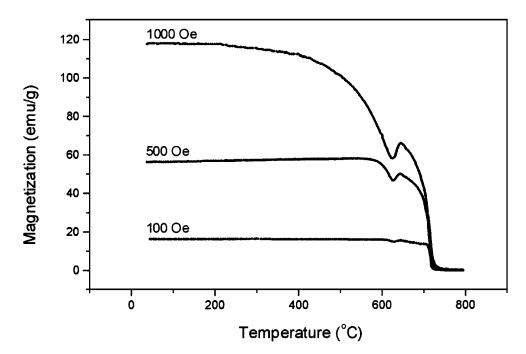


Figure 4 TMA heating curves of Fe-20Mo-5Ni-0.075C sample solution treated.

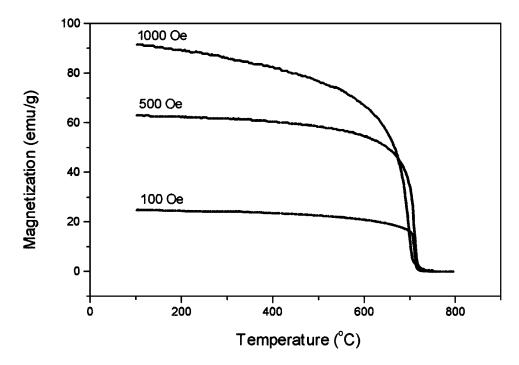


Figure 5 TMA cooling curves of Fe-20Mo-5Ni-0.075C sample solution treated.

since the values of B_r (14.4 kG) and $(BH)_{máx}$ (2.0 MG Oe) are maximum. For comparison, Table II lists these results and the properties of some commercial magnetic alloys with cobalt found in the literature [11]. As can be seen, only Vicalloy 2 presents properties comparable to those of the Fe-20Mo-5Ni-0.075C alloy.

3.2. X-ray diffraction

Figs 1, 2 and 3 show the X-ray diffraction spectra obtained in the samples solution treated, aged for 1 h and aged for 10 h, respectively. In the solution treated sample three peaks of ferrite α were found. The sample aged for 1 h present peaks of λ phase (Fe₂Mo) and/or R phase (Fe₆₃Mo₃₇). The sample aged for 10 h of aging present a small peak with interplanar distance d = 2.1416 Å, which suggests the presence of the carbide (Fe, Mo)₆C [8].

Also observed from the X-ray diffraction was a decrease of the ferrite parameter with increasing ageing time (Table III). This indicates an impoverishment of Mo and also C in the ferrite, during the aging, due to the precipitation of Mo-rich phases.

3.3. Thermomagnetic analysis

Fig. 4 presents the TMA heating curves for the solution treated sample, under three different applied fields: 100, 500 and 1000 Oe. The magnetic transitions are better observed in a higher applied field TMA

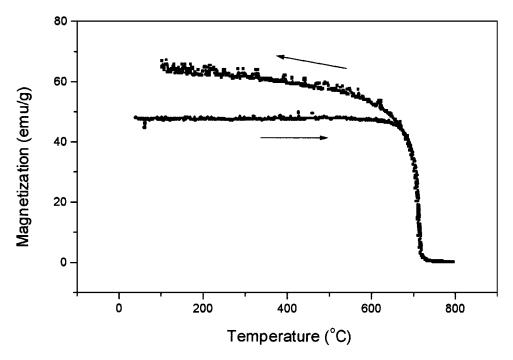


Figure 6 TMA curve of the sample previously aged at 610 °C for 4 h.

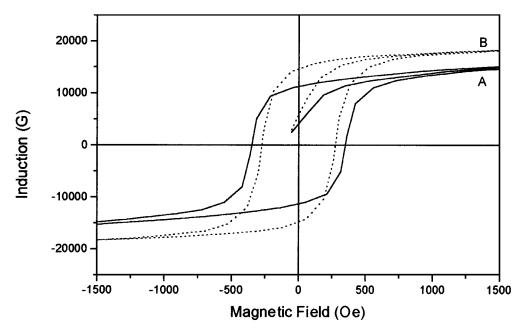


Figure 7 Hystereris loops before (B) and after (A) the TMA.

measurement. Two magnetic transitions (at 615 and 714 $^{\circ}\mathrm{C})$ were found.

The TMA cooling curves (Fig. 5), present only the magnetic phase transition at 714 °C. So, it's clear that the transition at 615 °C is irreversible. In fact, this is the Curie temperature of the ferrite supersaturated in molybdenum and carbon. The TMA thermal cycle acts as an ageing, in a manner that during the measurements, the precipitation of λ and/or R phases leads to the impoverishment of Mo in the α matrix. Thus, the phase with transition temperature at 615 °C corresponds to a rich molybdenum ferrite. When precipitation occurs and the ferrite becomes impoverished of Mo and C a new transition is then observed at 714 °C The first transition (615 °C) wasn't observed during the cooling because the supersaturated ferrite no more exist.

In samples previously aged at $610 \,^{\circ}$ C for 1 and 4 h (Fig. 6) only the transition at 714 $^{\circ}$ C is observed, which confirms that the phase with this transition temperature is a poor molybdenum ferrite found in the material after precipitation.

The precipitation which occurs during the TMA measurements causes a magnetic hardening, as can be seen in Fig. 7, by the comparison of the hysteresis loop before and after the TMA.

4. Conclusions

The magnetic properties (H_c , B_r and $(BH)_{máx}$) of the Fe-20Mo-5Ni-0.075C alloy are better than some cobalt containing commercial alloys (Remalloy 2, Vicalloy 1) and are comparable to those of Vicalloy 2.

During ageing treatments at 610 °C for 1 and 4 h, R and/or λ phases precipitate. After 10 h of ageing at 610 °C the (Fe, Mo)₆C carbide was also detected by X-ray diffraction. TMA curves show that the solution treated material undergoes two transitions, at 615 and 715 °C. The first transition (615 °C) is due to a supersaturated ferrite initially present in the material. During the TMA, the precipitation of R and/or λ phases takes molybdenum out of the solution. The second transition (714 °C) thus can be attributed to a poor molybdenum ferrite matrix.

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