The influence of thermo-mechanical treatment on the reverse martensitic transformation in Fe-30Ni alloy

F. Ciura[1]*, G. Michta[1], B. Dubiel[1], W. Osuch[1], Z. Jonšta[2], M. Tvrdý[2]

[1] *AGH University of Science and Technology, Faculty of Metals Engineering and Industrial Computer Science, Al. A. Mickiewicza 30, 30059 Krakow, Poland*
[2] *VSB Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, 17. listopadu 15, 70833 Ostrava-Poruba, Czech Republic*

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Abstract

The investigation of Fe-30Ni alloy, in which a martensite transformation can occur during plastic deformation or quenching in liquid nitrogen, was carried out. Application of magnetic methods allowed to determine characteristic temperatures of phase transformations and martensite volume fractions. Martensite produced during thermo-mechanical treatment was subjected to reversed transformation during subsequent annealing in a temperature range A_S–A_F. Results of microstructural analysis performed by light microscopy were correlated with mechanical properties determined in tensile tests.

K e y w o r d s: iron alloys, phase transformations, magnetic properties, tensile tests, light microscopy (LM)

1. Introduction

The Fe-30Ni alloy is of special interest of many research groups, also in the field of fundamental science, due to its properties and applications [1–5]. The alloy investigated exhibits single-phase austenitic structure at room temperature. Depending on thermomechanical treatment applied, the microstructure can be altered in wide range. In this alloy the martensitic transformation can occur both during quenching or plastic deformation. Martensite formed during plastic deformation has a different morphology from that achieved during quenching and forms so-called composite-like structure [6, 7]. During cold rolling in room temperature (RT) slip bands are formed at the monotonic deformation. A perpendicular rolling mode with the primary path realized in RT, and the secondary path rotated by 90° around primary longitudinal rolling direction leads to formation of transcrystalline shear bands. Decreasing the temperature down to −30 ℃ (below the temperature at which deformation induced martensitic transformation M_D begins) makes martensitic transformation to occur. The influence of thermo-mechanical treatment on the microstructure of Fe-30Ni alloy was described in detail in previous papers of the authors [8–10]. The subsequent annealing after martensitic transformation γ-α' leads to reverse martensitic transformation α'-γ. After repeated cooling in liquid nitrogen an inheritance of mechanical properties and microstructure morphology can be achieved [11–13].

In the present work the temperatures of phase transformations during cooling and heating of Fe-30Ni alloy were determined by magnetic methods. Volume fraction of martensite has been estimated for different thermomechanical treatments and after reverse transformation α'-γ. It was observed that a martensite volume fraction and morphology have a strong influence on the mechanical properties after reverse transformation.

2. Material and experimental procedures

The material investigated was a Fe-30Ni alloy. The detailed chemical composition is given in Table 1.

*Corresponding author: tel.: +48126172928; fax: +48126173190; e-mail address: ciura@agh.edu.pl

T a b l e 1. Chemical composition of the alloy investigated (wt.%)

C	Mn	P	S	Cu	Cr	Ni	Fe
0.01	0.11	0.007	0.013	0.04	0.38	28.5	bal.

T a b l e 2. The variants of thermo-mechanical treatment

A	cooling in liquid nitrogen
B	rolling at 20 ℃ with 30% strain perpendicular rolling at −30 ℃ with 30% strain cooling in liquid nitrogen
C	rolling at 20 ℃ with 30% strain perpendicular rolling at −60 ℃ with 30% strain cooling in liquid nitrogen
D	rolling at 20 ℃ with 30% strain perpendicular rolling at −80 ℃ with 30% strain cooling in liquid nitrogen

The alloy produced by vacuum melting was hot worked to obtain 8 mm thick sheets and annealed at 1150 ℃ for 1 hour. The alloy exhibited austenitic structure. For subsequent experiments 8 mm × 8 mm square specimens were cut off from the sheets. The variants of a thermo-mechanical treatment performed are given in Table 2. The specimens were subsequently annealed in a temperature range 350–600 ℃ in 30 ℃ intervals.

The temperatures of phase transformations (M_S, M_F, A_S and A_F) were determined by differential and torsion magnetometry. The measurements of magnetic permeability and saturation magnetization as the functions of temperature were performed.

The magnetization standard of the alloy was calculated on the basis of the results of X-ray measurements. The martensite and austenite volume fractions were determined by differential magnetometry. The detailed description of the magnetic methods is given elsewhere [14]. Microstructural analysis was conducted by means of light microscopy. Mechanical properties were measured in tensile tests.

3. Results and discussion

Changes of the magnetic permeability measured during cooling from 600 ℃ to −196 ℃ and subsequent heating up to 600 ℃ are shown in Fig. 1. At 600 ℃ the alloy exhibited austenitic structure. During cooling, martensitic transformation has started at temperature around $M_S = -90$ ℃ and finished at approximately $M_F = -170$ ℃.

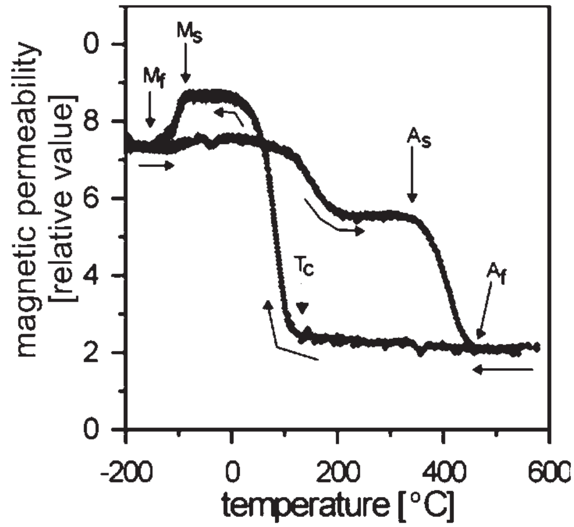

Fig. 1. The changes of magnetic permeability during cooling and heating of Fe-30Ni alloy.

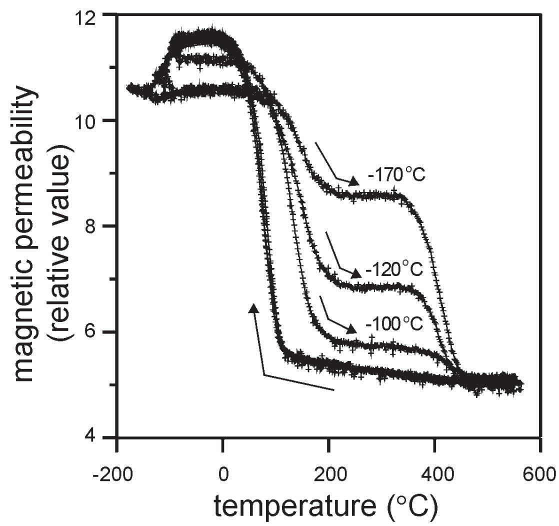

Fig. 2. The changes of magnetic permeability during cyclic cooling and heating of Fe-30Ni alloy up to −100 ℃, −120 ℃ and −170 ℃.

The Curie temperature T_C of austenite is around 100 ℃ and therefore the increase in magnetic permeability is observed. The stresses produced below M_S temperature influence a decrease of permeability, although the martensite formed is ferromagnetic. Such tendency is observed until M_F temperature is reached. During subsequent cooling down to the temperature of liquid nitrogen no microstructural changes occur, what results in the constant value of the permeabil-

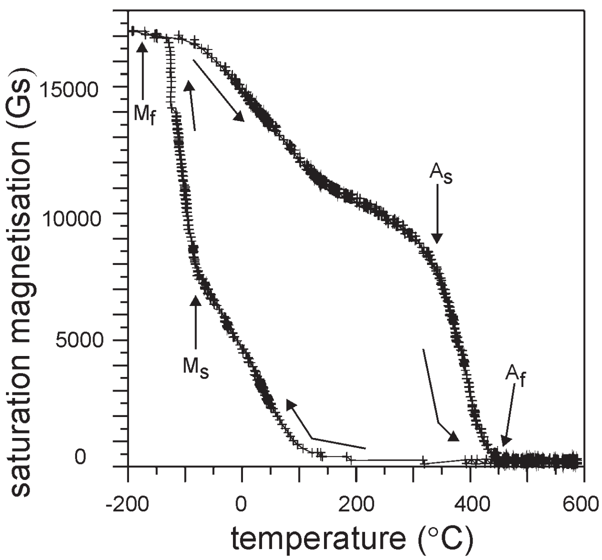

Fig. 3. The changes of magnetic saturation during cooling and annealing.

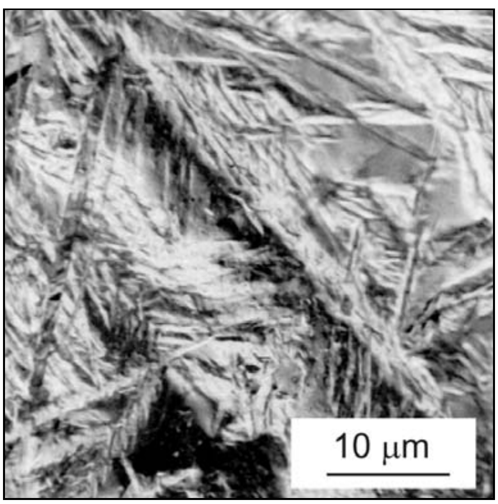

Fig. 4. Microstructure after perpendicular rolling at room temperature.

ity. The following heating up to 600 ℃ results in a decrease of magnetic permeability near 140 ℃ and in the temperature range 350–450 ℃. Near 140 ℃, the effect is connected with the Curie temperature of retained austenite. In the temperature range 350–450 ℃ a pronounced decrease of magnetic permeability is related to the transformation of ferromagnetic martensite into paramagnetic austenite. It can be therefore concluded that the temperature of beginning and finish of reverse martensite transformation is $A_{\mathrm{S}} = 350$ ℃ and $A_{\mathrm{F}} = 450$ ℃, respectively.

The changes of magnetic permeability during cyclic cooling and heating up to −100 ℃, −120 ℃ and

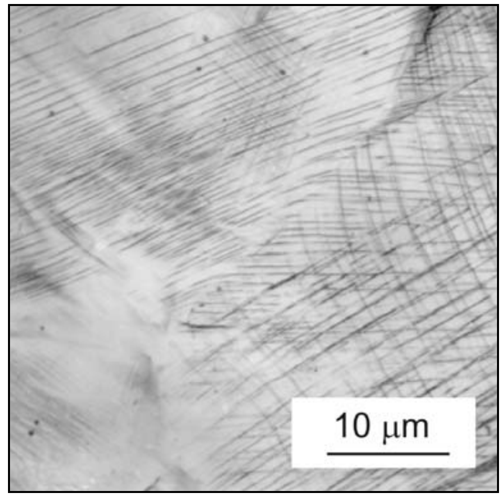

Fig. 5. Microstructure after cooling according to variant A.

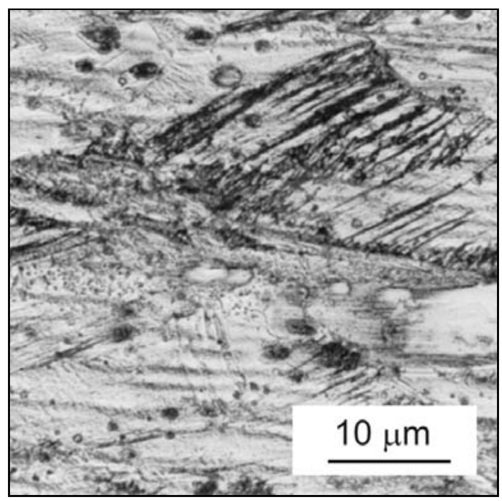

Fig. 6. Microstructure after deformation according to variant B, before cooling in liquid nitrogen.

−170 ℃ (Fig. 2) can be correlated with the influence of temperature on the amount of martensite in a M_{S}–M_{F} range. During cooling from 600 ℃ to −90 ℃ and subsequent heating up to 260 ℃ the changes in magnetic permeability are connected only with a hysteresis of Curie temperature of austenite. Decreasing the temperature down to −100 ℃ leads to the formation of a small amount of martensite. This phenomenon is observed as a new component of magnetic permeability. At lower temperatures magnetic permeability increases with increasing of the amount of martensite. In the temperature range 350–450 ℃ the magnetic effect disappears and this can be related with the reverse martensitic transformation.

To verify the determined values of M_{S}, M_{F}, A_{S}, A_{F} temperatures, the measurements were repeated with

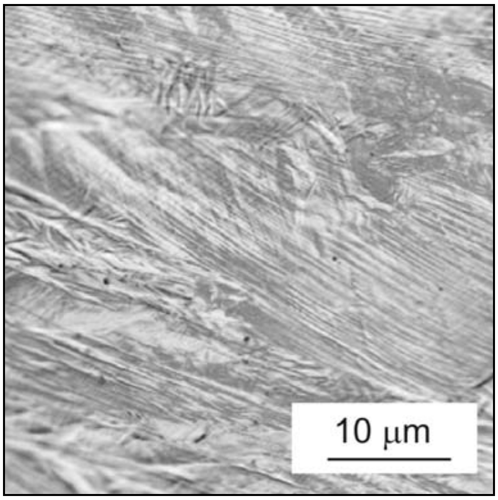

Fig. 7. Microstructure after deformation according to variant B.

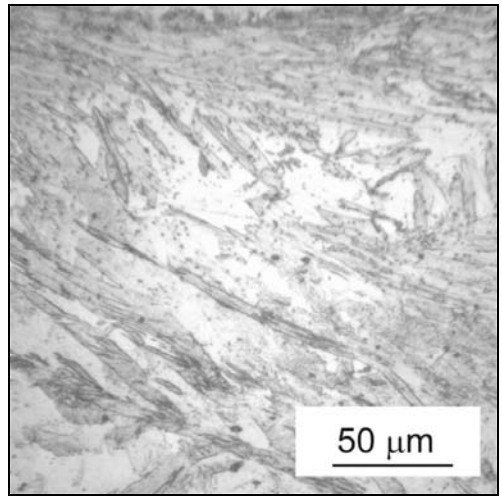

Fig. 9. Microstructure after deformation according to variant D.

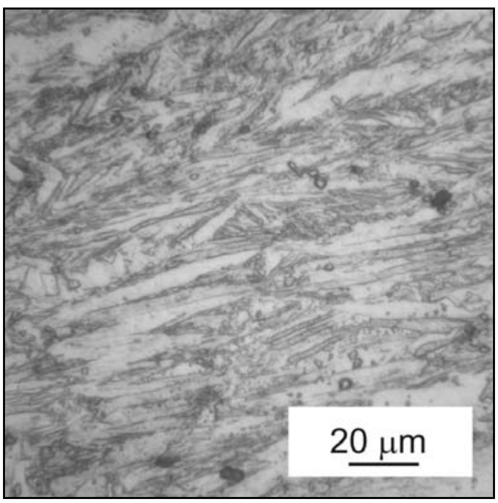

Fig. 8. Microstructure after deformation according to variant C.

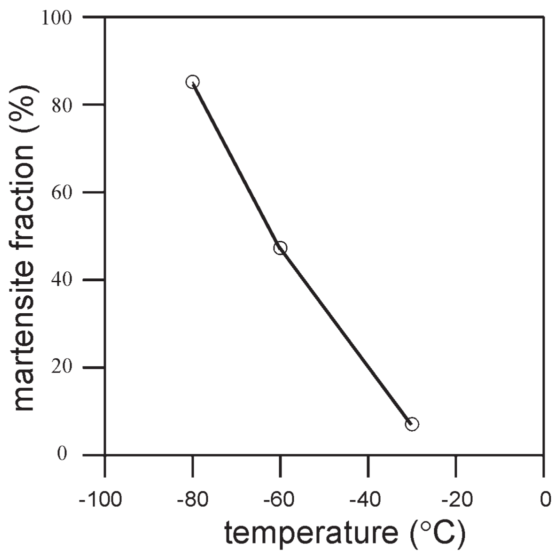

Fig. 10. Martensite volume fractions after deformation according to variants B, C, D.

the use of a torsion magnetometer. Figure 3 shows the changes of saturation magnetization detected during cooling and heating. The results obtained by both differential and torsion magnetometry are in a good agreement.

Microstructural analysis allowed the observation of different martensite morphologies formed after thermo-mechanical treatments. Perpendicular rolling at room temperature with 30% strain leads to transcrystalline shear bands formation (Fig. 4). After cooling in liquid nitrogen according to variant A the martensitic morphology is similar as in a quenched steel (Fig. 5).

The decrease in the temperature of second perpendicular rolling down to −30 ℃ (according to variant B, before cooling in liquid nitrogen) leads to the formation of composite-like structure consisting of transcrystalline martensite plates aligned on the previous shear bands in austenite (Fig. 6). After subsequent cooling in liquid nitrogen (variant B) the martensite transformation completes and the martensite morphology is similar to that obtained in the variant A (Fig. 7). At lower temperature of second rolling, i.e. at −60 ℃ and −80 ℃ (variants C and D, respectively) the amount of the deformation induced martensite increases, as shown in Figs. 8 and 9.

The values of the martensite volume fraction after rolling at −30 ℃, −60 ℃ and −80 ℃ determined by magnetic methods are given in the diagram presented in Fig. 10.

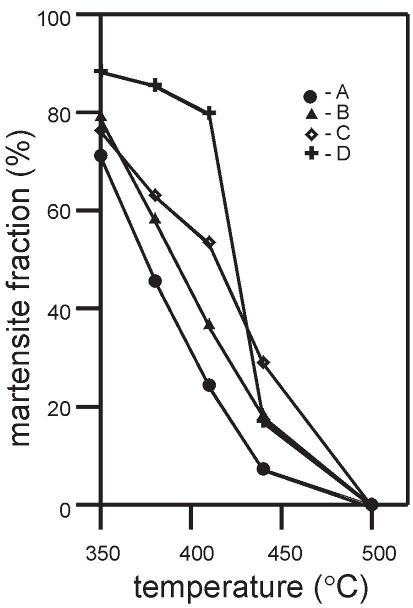

Fig. 11. Martensite volume fractions after reverse transformation α'-γ.

It can be concluded that the amount of the martensite depends very strongly on temperature.

Figure 11 shows the diagram of martensite volume fraction after reverse transformation α'-γ in the temperature range 350–500 ℃. The annealing was performed at 30 ℃ step for 10 minutes for specimens deformed according to variants A, B, C and D. The amount of martensite decreased with increasing temperature.

Tensile tests were performed for specimens deformed according to variants A and B. The results of the tensile tests and martensite volume fraction measurements are listed in Table 3.

The analysis of the results leads to the conclusion that the martensite volume fraction and morphology influence the mechanical properties after the reverse transformation α'-γ.

4. Conclusions

1. Cooling of Fe-30Ni alloy down to temperature −90 ℃ and subsequent annealing up to 600 ℃ leads to the hysteresis of magnetic properties of austenite.
2. The determined temperatures of the start and finish of reverse martensite transformation in Fe-30Ni alloy are $A_S = 350$ ℃ and $A_F = 450$ ℃, respectively.
3. Martensite volume fraction and morphology influence the mechanical properties after reverse transformation α'-γ.

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Table 3. The results of tensile tests and corresponding martensite volume fractions for specimens deformed according to variants A and B and subsequently annealed at 350 ℃, 380 ℃, 410 ℃, 440 ℃ and 600 ℃ for 10 minutes

Nr	Temperature of annealing (℃)	Variant	R_m (MPa)	$R_{0.2}$ (MPa)	A_R (%)	A_5 (%)	Martensite volume fraction (%)
1	350	A	814	728.9	2.9	7.1	82
2		B	916.2	799	1.7	5	87
3	380	A	759.5	670.6	3.0	7.3	45.6
4		B	881.8	739	1.7	5.9	58.1
5	410	A	590.8	512.2	5.2	9.7	24.4
6		B	830.1	731	2.3	7.7	36.5
7	440	A	581.8	475.9	7.3	11.7	7.3
8		B	784.4	682.1	2.3	9.5	18.1
9	600	A	542.1	462.7	9.0	15.0	0
10		B	592	493.9	5.7	11.9	0

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