Letter to the Editor

The influence of thermomechanical treatment on structure and properties of Fe-30Ni alloy

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Abstract

The material investigated was Fe-30Ni (wt.%) alloy with various morphologies and volume fractions of martensite induced by plastic deformation or quenching in liquid nitrogen. The characteristic temperature of phase transformations, especially the start temperature of martensitic transformation M_S, was determined using magnetic measurements. It was found that M_S is affected by the grain size of the alloy. The use of thermo-mechanical treatment leading to martensitic transformation followed by annealing above the start temperature of reversed transformation resulted in refining of the alloy microstructure and improving its strength and ductility.

K e y w o r d s: iron alloys, deformation structure, reversed martensitic transformation, light microscopy (LM), transmission electron microscopy (TEM), rolling, tensile tests

1. Introduction

Depending on the content of nickel and carbon, the microstructure of Fe-Ni alloys can be pearlitic, martensitic or austenitic. The most popular is Fe-30Ni (wt.%) alloy, with austenitic structure at room temperature (RT), which can be transformed into martensite [1, 2]. The morphology and volume fraction of martensite can be controlled in wide range by changing the strain, temperature and deformation path [3]. This enables to achieve the unique combination of structure, texture and property characteristics of composite-like materials [4, 5]. Similar investigations with the use of thermo-mechanical treatment in fabrication of composite-like metallic materials were performed on steels and brasses [6].

In Fe-30Ni (wt.%) alloy the strengthening phase is deformation-induced martensite with directionally aligned transcrystalline martensite needles within austenite. However the increased strength of this material is accompanied with deterioration of ductility. The problem of high strength and ductility of materials was investigated by many research groups. For example Talonen et al. [7] investigated the influence of the deformation rate on the $\gamma \rightarrow \alpha$ transformation in steels. They have found that the changes of the martensite fraction influence strongly the strain rate, tensile strength and ductility.

Jin and Young-Kook Lee [8] have achieved high strength and ductility in austenitic steels by conventional rolling and annealing. After homogenization and hot rolling the steel was deformed at room temperature, what induced martensitic transformation. During annealing above the start temperature of reversed transformation, fine-grained microstructure was achieved. The repeated rolling and annealing led to metastable austenitic structure with grain size of 200 μm. Such deformed material exhibited high strength and good ductility.

In the present work, perpendicular rolling of Fe-30Ni (wt.%) alloy in temperature range M_S–M_F resulted in strengthening due to the phase transformation $\gamma \rightarrow \alpha'$. After subsequent cooling in liquid nitrogen and annealing above the start temperature of

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Table 1. Chemical composition of the investigated alloy (wt.%)

Fe	Ni	C	Mn	P	S	Cu	Cr
Bal.	28.5	0.01	0.11	0.07	0.013	0.04	0.38

reversed transformation M_R, a fine-grained austenitic structure was achieved. The annealing temperature was chosen to be not too high to avoid the grain growth. To enhance the process of grain refining, cooling and annealing was repeated three times.

2. Material

Chemical composition of the investigated material is given in Table 1.

The alloy produced by vacuum melting was hot worked to achieve 8 mm thick sheets and annealed at 1150 ℃ for 1 hour. The alloy exhibited austenitic structure. The following variants of thermomechanical treatment were performed to achieve a composite-like microstructure:

Variant I

A. Rolling with 30 % reduction at 20 ℃ + perpendicular rolling (with the same rolling direction) with 30 % reduction at –30 ℃

B. The same as A + cooling in liquid nitrogen

C. The same as B + annealing at 500 ℃ for 15 minutes (to obtain reversed transformation)

D. The same as C + cooling in liquid nitrogen

Variant II

E. Rolling with 30 % reduction at RT + perpendicular rolling with 30 % reduction at –80 ℃

F. The same as E + cooling in liquid nitrogen

G. The same as F + annealing at 550 ℃ for 15 minutes

H. The same as F + triple annealing and cooling.

For all variants, microstructural investigations and tensile tests were performed. Microstructure characterization was conducted by light microscopy (LM) and analytical transmission electron microscopy (TEM). Phases were identified by selected area electron diffraction patterns.

Tensile tests were performed using cylindrical specimens with 3 mm diameter and gauge length to diameter ratio 5 : 1. The strain rate of $2 \times 10^{-3}\,s^{-1}$ was applied. The values of tensile strength R_m, yield strength $R_{0.2}$, total elongation A_5, and uniform elongation A_r were determined.

Specimens deformed and cooled in liquid nitrogen were subjected to magnetic measurements to determine temperatures of phase transformations, especially start temperature of martensitic transformation M_S.

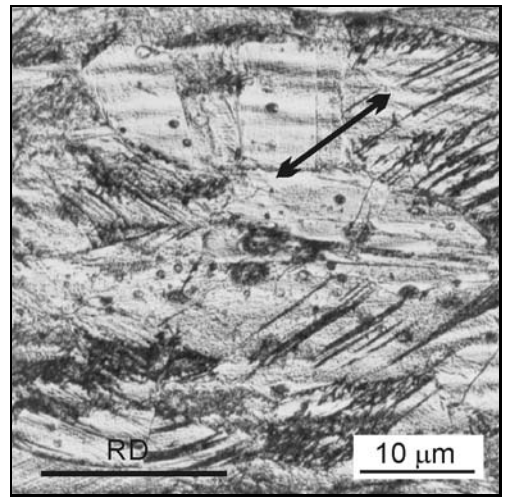

Fig. 1. Microstructure after perpendicular rolling with 30 % reduction at −30 ℃ according to Variant I A (LM).

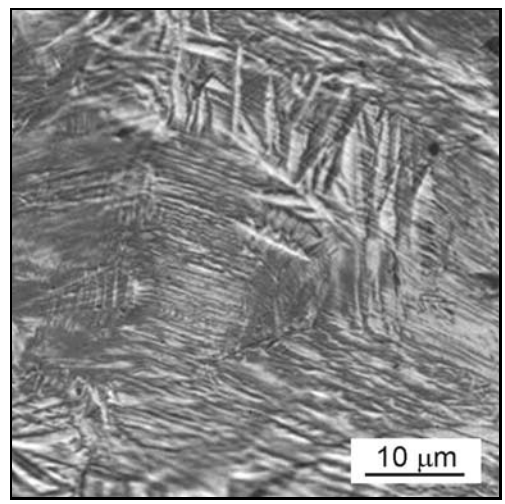

Fig. 2. Microstructure after perpendicular rolling with 30 % reduction at −30 ℃ and cooling in liquid nitrogen according to Variant I B (LM).

3. Results and discussion

Detailed description of the microstructure formed after Variant I was given in [4, 9].

Perpendicular rolling with 30 % reduction below start temperature of deformation induced martensitic transformation $M_D = -30$ ℃ leads to the formation of composite-like martensite (Fig. 1). When the transformation is completed during cooling in liquid nitrogen, the formed martensite is similar like that in the quenched steel (Fig. 2).

After reversed transformation, no recrystallized austenitic structure is observed on TEM images (Fig. 3).

Table 2. Tensile properties of the specimens deformed according to Variant I

Deformation scheme	R_m (MPa)	$R_{0.2}$ (MPa)	A_5 (%)	A_r (%)
A	732	635	10.0	2.2
B	895	687	11.7	5.1
C	621	499	14.2	4.9
D	893	699	13.2	5.8

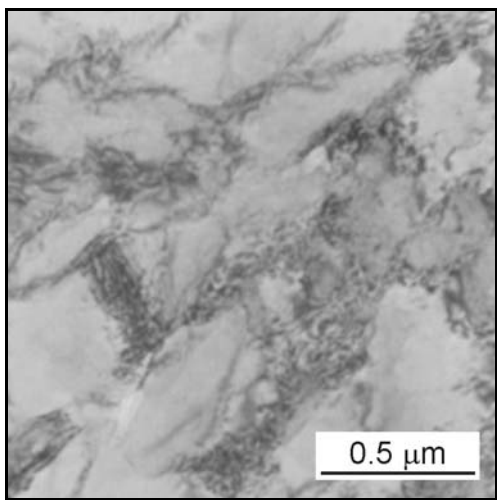

Fig. 3. TEM image of the microstructure after reversed transformation (annealing at 500 °C for 5 minutes).

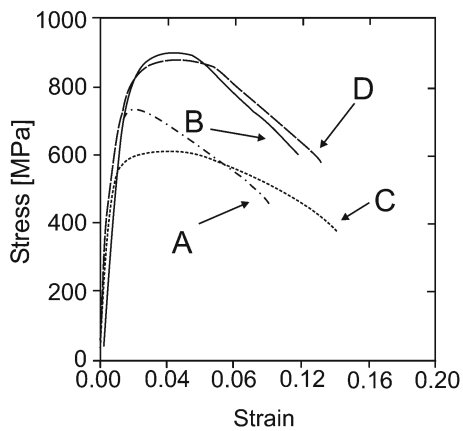

Fig. 4. Tensile curves of specimens deformed according to Variant I A–D.

Short annealing at 500 °C, above the start temperature of reversed transformation, and subsequent cooling in liquid nitrogen, result in inheritance of the material's structure and properties [5, 10].

Tensile curves of the specimens deformed according to Variant I A–D are shown in Fig. 4.

For specimens B and D, the curves are very similar, however, specimen D exhibits higher ductility. The mechanical properties determined from tensile tests

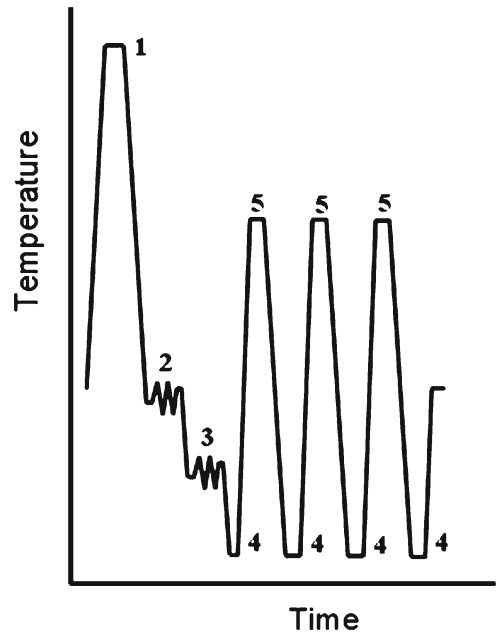

Fig. 5. Schematic diagram of Variant II thermo-mechanical treatment: 1 – annealing at 1150 °C/1 h, 2 – rolling with 30 % reduction at RT, 3 – perpendicular rolling with 30 % reduction at –80 °C, 4 – cooling in liquid nitrogen, 5 – annealing at 500 °C/5 min.

are given in Table 2.

On the basis of these results the experiments in variant II were performed [11].

Schematic diagram of the thermomechanical treatment is shown in Fig. 5. In order to increase volume fraction of martensite the rolling was performed at a temperature of −80 °C, close to M_S. The achieved fine-grained microstructure is shown in Fig. 6. TEM investigations show the fine grains within the needle of primary austenite (Fig. 7). Identification of martensite and austenite after such thermomechanical treatment is very difficult because of high density of defects in austenite. In the present work the austenite grains were identified by means of electron diffraction (SAED) and TEM dark field imaging. Figure 8 shows the two-phase martensitic and austenitic structure of specimen deformed according to Variant II 4 in bright- (Fig. 8a) and dark-field (Fig. 8b) images. Corresponding SAED pattern is shown in Fig. 8c and schematically in Fig. 8d.

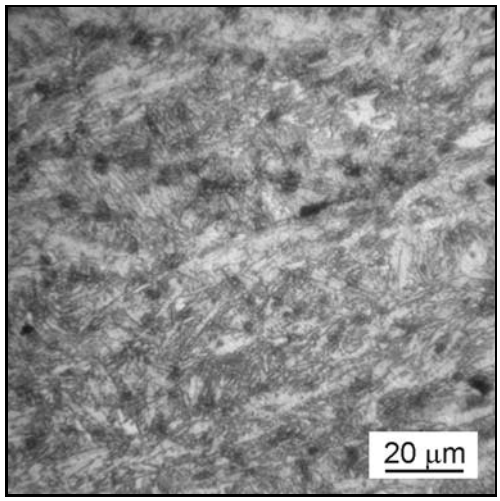

Fig. 6. Microstructure after cyclic treatment according to Variant II H.

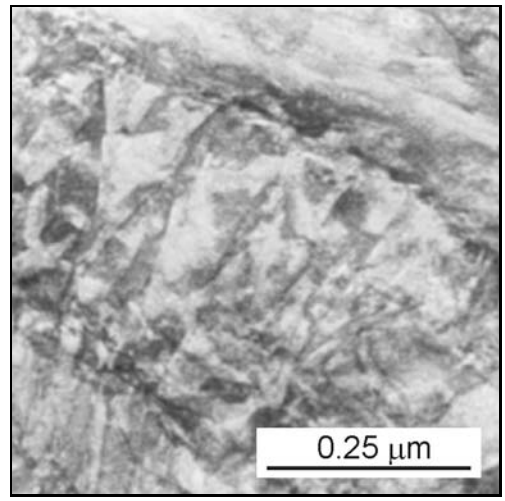

Fig. 7. Microstructure of the specimen deformed according to Variant II (TEM).

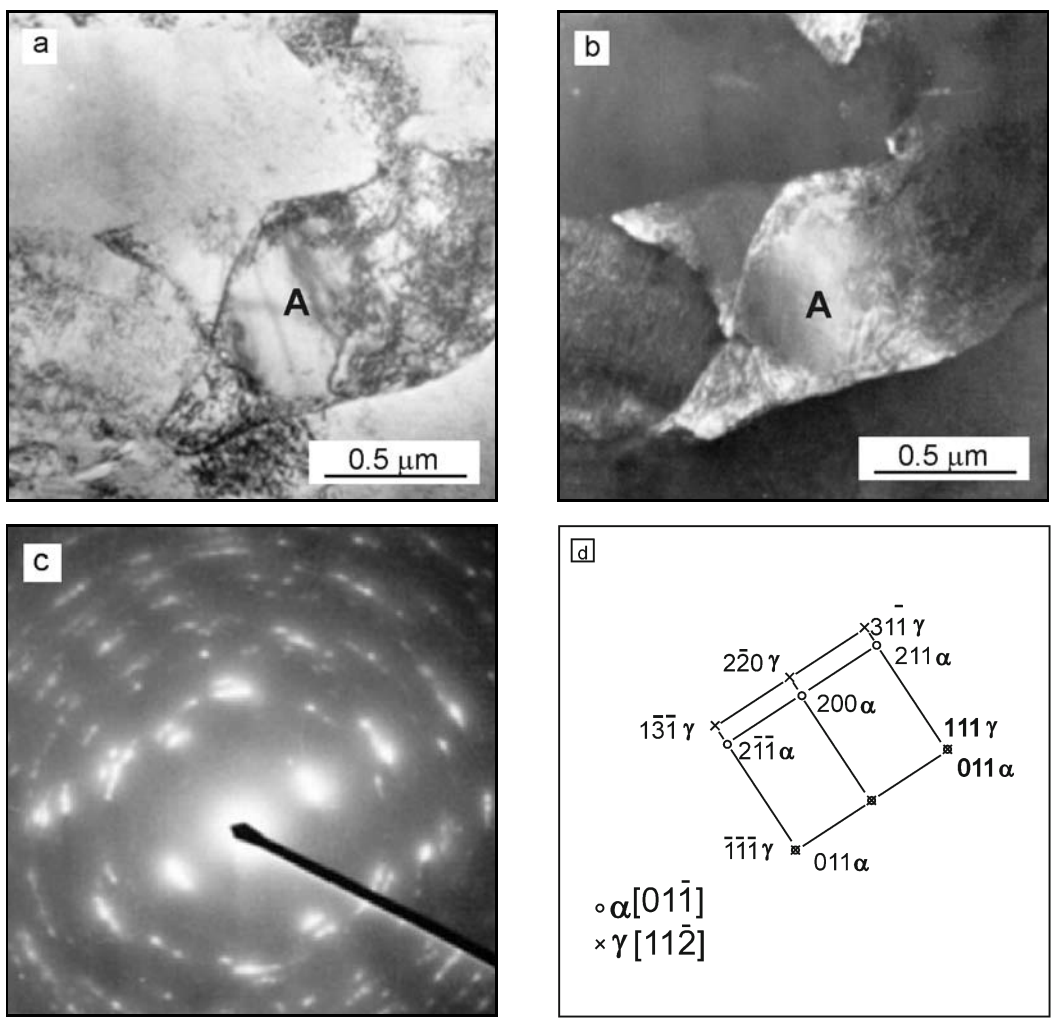

Fig. 8. Two-phase (martensite, austenite) alloy microstructure after Variant II 4: a) TEM bright-field, b) TEM dark-field image, c) SAED pattern, d) identification of SAED pattern.

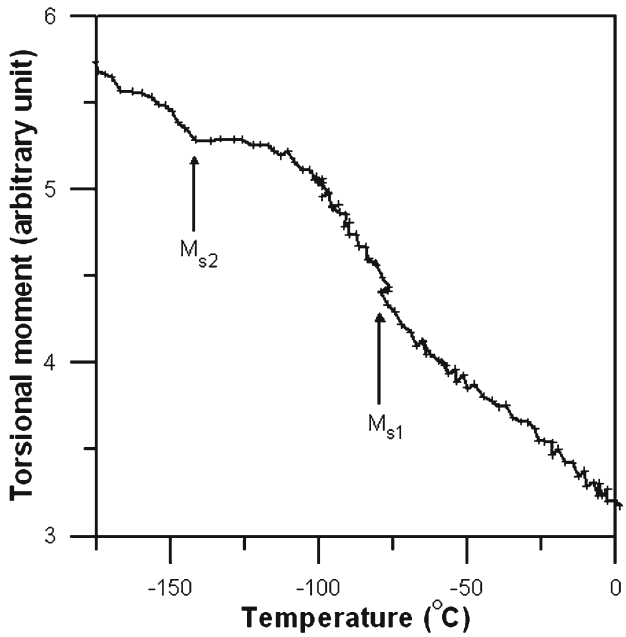

Fig. 9. Changes of torsion moment during annealing and cooling of specimens deformed according to Variant II.

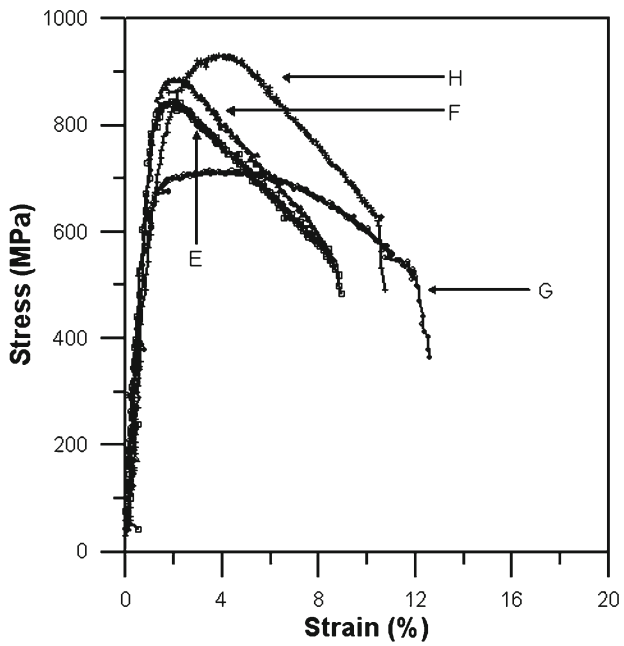

Fig. 10. Tensile curves of the specimens deformed according to Variant II E–H.

The results of measurements of magnetic permeability during cooling and annealing in temperature range −197 ℃ – 600 ℃ were described in detail in [9]. The M_S temperature was measured as to be −90 ℃ [12]. The changes of torsion moment during cooling after cyclic treatment according to Variant II D are shown in Fig. 9.

Two inflection points corresponding to M_{S1} and M_{S2} temperatures were found on curves. Observation of two M_S temperatures might be connected with the presence of two austenite fractions with different density of structural defects. The first austenite fraction, formed after cyclic thermo-mechanical treatment, exhibits pronounced grain refinement and high density of dislocations. The second fraction of austenite is formed during slow cooling in air after annealing in the last step of cyclic treatment. After strong deformation during slow cooling from temperature of 500 ℃, the recovery process can appear.

Therefore it can be concluded that M_{S1} is connected with the start of austenite-martensite transformation of the recovered austenite with lower dislocation density.

The lower temperature $M_{S2} \sim -140$ ℃ is probably connected with the transformation of the highly defected austenite between martensite needles.

To verify this hypothesis, the additional experiment was performed to determine M_S temperature after solution treatment and deformation with 40 % reduction. It was found, that for both variants the difference in M_S temperature was 15 ℃.

The M_S temperature strongly depends on the grain size. Magnetic measurements performed in previous works [9, 12] have shown, that the M_S temperature of specimens treated according to Variant I was −90 ℃. The mean grain diameter after this variant was equal to 6 μm.

In specimens annealed at 835 ℃, in which the grain growth appeared and the mean grain diameter was 15 μm, the M_S temperature was −58 ℃ [13]. The martensite needles are formed within austenite grains and their size corresponds to an initial austenite grain size. It can be concluded, that thermo-mechanical treatment leading to grain refinement decreases M_S. Phase transformations cause the increase of strengthening. Non-transformed austenite is highly deformed (contains a high dislocation density) and affects alloy plastic properties.

Figure 10 shows the tensile curves of specimens deformed according to Variant II. The tensile properties are listed in Table 3.

4. Conclusions

1. The fine-grained microstructure of Fe-30Ni (wt.%) alloy can be achieved by cyclic thermo-mechanical treatment.

2. The grain refinement influences decreasing of the M_S temperature of the alloy.

3. The strength and ductility of Fe-30Ni (wt.%) alloy can be improved by its microstructure refinement by means of cyclic thermo-mechanical treatment.

Table 3. Tensile properties of the specimens deformed according to Variant II

Deformation scheme	R_m (MPa)	$R_{0.2}$ (MPa)	A_s (%)	A_r (%)
1	873	799	9.3	1.8
2	889	842	8.9	2.2
3	651	599	12.7	4.3
4	929	781	10.2	4.1

Acknowledgements

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