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X-ray diffraction and magnetic analysis of deformation induced martensites in a Fe-17Mn-1.9Al-0.1C steel

S. S. M. TAVARES
Laboratoire de Cristallographie, CNRS BP 166, 38042 Grenoble Cedex 9, France;
Universidade Federal Fluminense—Depto. de Engenharia Mecânica, R. Passo da Pátria, 156, CEP 24210-470, Niteroi, Brasil

A. LAFUENTE
Laboratoire de Cristallographie, CNRS BP 166, 38042 Grenoble Cedex 9, France;
Instituto de Ciencias de los Materiales de Aragón, CSIC—Universidad de Zaragoza, 50009 Zaragoza, Spain

S. MIRAGLIA, D. FRUCHART
Laboratoire de Cristallographie, CNRS BP 166, 38042 Grenoble Cedex 9, France

The deformation induced martensitic transformations in a Fe-17Mn-1.9Al-0.1C steel are investigated by X-ray diffraction and magnetic measurements. Martensites α' and ε are formed at the same time during cold deformation of the steel. The starting (A_s) and final (A_f) temperatures of the reversion of martensite α' during continuous heating are 390°C and 540°C. The martensite ε seems to be reverted in the 300°C–450°C range. These results are discussed and compared with those previously obtained in conventional stainless steels.

1. Introduction

FeMnAl alloys have been extensively studied in recent years due to their promising mechanical and corrosion resistance properties [1–3].

Our interest is concentrated in a group of FeMnAl alloys which is austenitic in the solution treated condition and may undergo a martensitic transformation induced by cold deformation. In the work of Chen *et al.* [4, 5] a Fe-20.5Mn-1.1Al-0.1C (wt%) alloy was partially transformed into martensite during deformation by rolling and/or bending. According to the authors, the martensite formed was body centered cubic and ferromagnetic. When the Al content was increased to 2.0%, the martensitic transformation was suppressed.

A similar behavior can be found in the austenitic stainless steels, that are fully austenitic and paramagnetic after a solution treatment at around 1000°C and may present martensites α' (bcc, ferromagnetic) and/or ε (hcp, paramagnetic) induced by deformation, hydrogen charging or ion irradiation. In metastable steels (ex.: AISI 301, 302, 304, 304L, ...) martensites ε and α' may be produced by cold deformation. Since martensite ε is found in small deformation range ($r < 15\%$) and martensite α' the predominant for higher deformations, many authors [6–9] reported the sequence of transformation as being $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. In stable steels (ex.: AISI 310) only martensite ε can be formed by plastic deformation.

It's also of our interest the stability of the martensite phases. Some researchers investigated the reversion of deformation induced martensites during heating in stainless steels [9–12]. In the study of Singh [9], the ε and α' martensites of an AISI 304 steels were stable up to 473 K and 673 K respectively. In our recent study [10] of the α' reversion in an AISI 304 steel, the starting (A_s) and final (A_f) temperatures of the reversion varied from 433°C to 445°C and from 705°C to 724°C depending on the amount of deformation and heating rate.

In the present work, the deformation induced martensitic transformations and their reversion in a Fe-17Mn-1.9Al-0.1C (wt%) alloy were investigated by means of X-ray diffraction and magnetic measurements. The results are discussed and compared to those found in conventional stainless steels.

2. Materials and methods

A Fe-17Mn-1.9Al-0.1C (wt%) alloy was melted in an arc furnace from high purity materials. The chemical composition of the material was checked by plasma spectroscopy (Table I).

The ingot was hot forged with a high-speed hammer at 1000°C. The material was solution treated at 1050°C during two hours in vacuum and then water quenched. The plate (3.86 mm. thick) was rolled at room temperature to different degrees of thickness reduction (r) and true strain (ε_1): $r = 27.5\%$ ($\varepsilon_1 = -0.32$), $r = 54.4\%$ ($\varepsilon_1 = -0.785$), $r = 74.1\%$ ($\varepsilon_1 = -1.35$) and $r = 94.8\%$ ($\varepsilon_1 = -2.96$). After deformation, some samples were

TABLE I Chemical composition of the material (wt%)

%Mn	%Al	%C	%S	%Fe
17.13	1.89	0.10	0.004	Balance

heat treated in the 100–800°C range in order to evaluate the martensitic phase's stability. The stability of martensite α' was also evaluated by thermomagnetic analysis (TMA) of the sample deformed with $r = 74.1\%$ ($\varepsilon_1 = -1.35$).

X-ray diffraction was carried out in a Phillips diffractometer using Kα Cu radiation. The TMA was carried out in a thermomagnetic balance with the sample sealed in a quartz tube under vacuum. The heating rate used in the TMA was 10°C/min.

3. Results and discussion

Fig. 1 shows the diffractogram of the sample solution treated. At this condition, the material is paramagnetic and presents a fcc structure with parameter $a = 3.588$ Å. Fig. 2 shows the diffractograms of the samples deformed with $r = 27.5\%$ ($\varepsilon_1 = -0.32$), $r = 54.4\%$ ($\varepsilon_1 = -0.785$) and $r = 94.8\%$ ($\varepsilon_1 = -2.96$). The cold deformation induces the formation of martensites ε and α'. Peaks of these two phases were found in all deformed samples.

Fig. 3 shows that the most intense peak in the diffractograms of Fig. 2 may be decomposed in three reflections: γ_{111} ($d = 2.070$ Å), ε_{002} ($d = 2.055$ Å) and α'_{110} ($d = 2.026$ Å). The peak separation was performed using the software WINPLOTR® for X-ray diffraction analysis. A comparison between curves (a) and (b) suggests that the increase of deformation promotes a reinforcement of the ε_{002} in detriment of α'_{110} and γ_{111}. On the other hand, the magnetic measurements (Fig. 4) show that the saturation magnetization increases with deformation. It could mean that the amount of the magnetic phase α' increases, but in this case this result would be discrepant with the one obtained by X-ray diffraction. We consider two possibilities to explain this discrepancy:

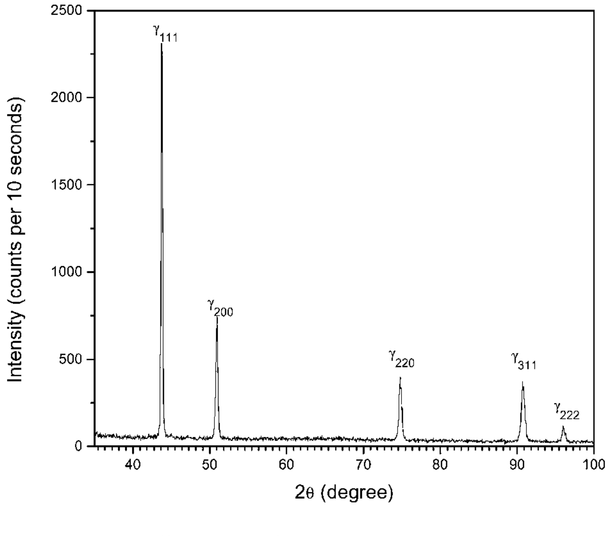

Figure 1 X-ray diffractogram of the solution treated sample.

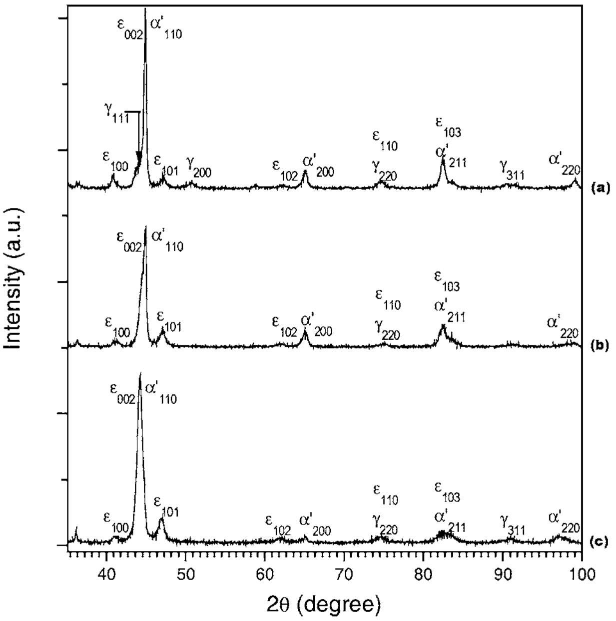

Figure 2 X-ray diffractograms of deformed samples: (a) $r = 27.5\%$ ($\varepsilon_1 = -0.32$); (b) $r = 74.1\%$ ($\varepsilon_1 = -1.35$) and (c) $r = 94.8\%$ ($\varepsilon_1 = -2.96$).

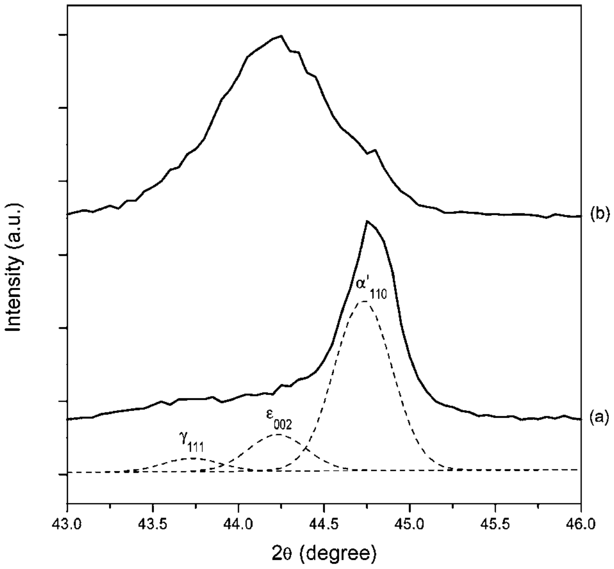

Figure 3 Detail of the X-ray diffractograms of deformed samples: (a) $r = 27.5\%$ ($\varepsilon_1 = -0.32$) and (b) $r = 94.8\%$ ($\varepsilon_1 = -2.96$). The peak of diffractogram (a) was decomposed in three reflections: γ_{111}, ε_{002} and α_{110}.

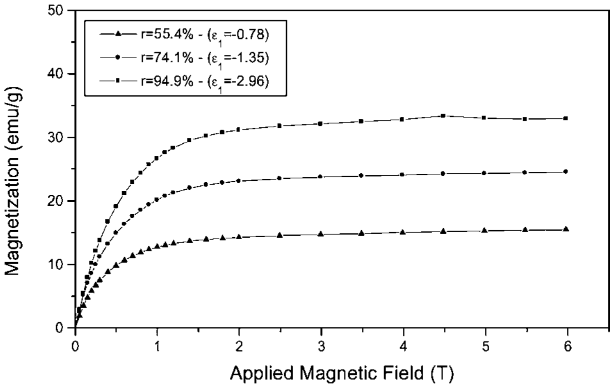

Figure 4 Magnetization curves of the samples deformed with $r = 55.4\%$ ($\varepsilon_1 = -0.78$), 74.1% ($\varepsilon_1 = -1.35$) and 94.9% ($\varepsilon_1 = -2.96$).

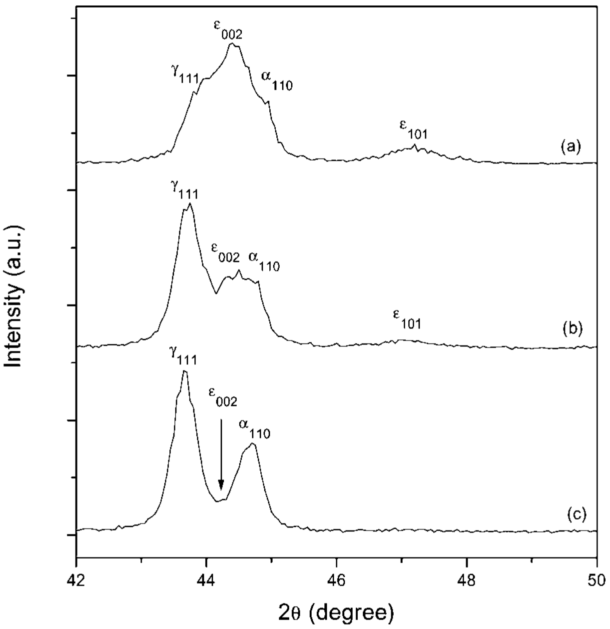

Figure 5 Diffractograms of samples deformed and heat treated at (a) 300°C, (b) 350°C and (c) 400°C for 1 hour.

- the decrease of the amount of phase α' suggested by Fig. 3 is rather an effect of texture, and, in this case, the amount of phase α' increase accordingly to the saturation magnetization results; or
- the increase of magnetization with deformation is rather due to a magnetostriction effect on phase α' than to an increase of the amount of this phase. It means, the magnetism of α' phase may be affected by strain and residual stresses.

This point is not yet clarified, and these two possibilities must be investigated. Nevertheless, is clear that the sequence of transformation $\gamma \to \varepsilon \to \alpha'$ observed in the common stainless steels is not observed in these Fe-17Mn-1.9Al-0.1C alloy, since the two martensitic phases (ε and α') are present in all deformed samples.

Two peaks that appeared in Fig. 2 couldn't be identified: $d = 1.570$ Å ($2\theta_1 = 58.75°$) and $d = 2.481$ Å ($2\theta_2 = 36.17°$). These two peaks may form another cubic phase (φ) with $a_\varphi = 4.962$ Å, which is related to the α' latice parameter by $a_\varphi = a_{\alpha'}\sqrt{3}$. In fact, φ is a superstructure of the α' phase that was created by the deformation process.

In the samples heated after deformation, no modification was found until 300°C. Fig. 5 shows that it is between 300°C and 350°C that the phase ε starts to revert into austenite, since the ε_{101}, ε_{102} and ε_{002} reflections become less intense. Between 350°C and 400°C the peaks ε_{101} and ε_{102} disappear, but the ε_{002} reflection seems to be still present between the α_{110} and the γ_{111} peaks. This ε reflection is then eliminated by an annealing at 450°C (not shown). So, we consider the range of the reversion of martensite ε as being from 300°C to 450°C. The martensite ε produced by deformation in the Fe-17Mn-1.9Al-0.1C alloy is reverted in a temperature range higher than that found in stainless steels.

When the steel is heat treated in the 450–750°C range large amounts of ferrite (α) are formed. It happens by diffusion mechanism since the reaction $\gamma \to \alpha$ is predicted in the Fe-Mn equilibrium phase diagram. Fig. 6 compares the X-ray diffractograms of samples heat treated at 700°C, 750°C and 800°C for 1 hour after deformation. As can be seen, is only after a heat treatment at 800°C that a fully austenitic structure is obtained. From Fig. 6, the temperature that delimits the $\alpha + \gamma$ and γ fields (A_3 temperature) must placed between 750°C and 800°C, which is at least 200°C above the value predicted in the Fe-Mn phase diagram [13] for a Fe-17Mn alloy. This increase of A_3 is due to the α-stabilization effect of aluminum.

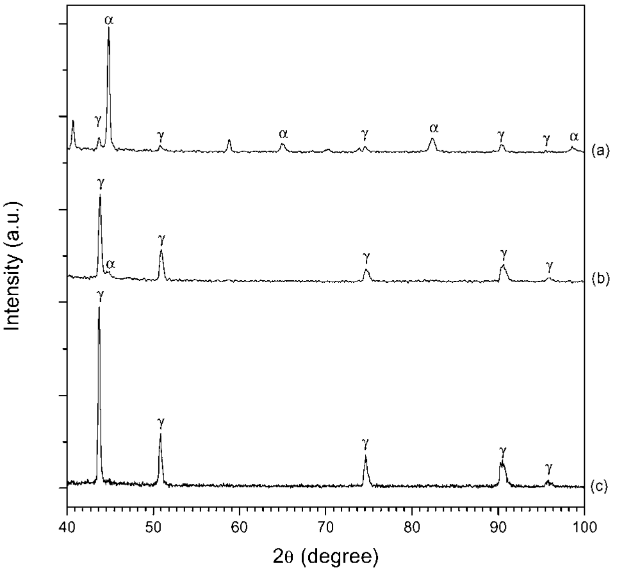

Figure 6 Diffractograms of samples deformed and heat treated at (a) 700°C, (b) 750°C and (c) 800°C for 1 hour.

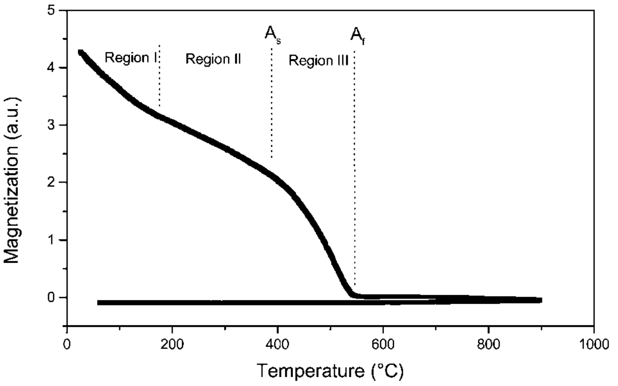

Figure 7 TMA curve of the sample previously deformed with $r = 74.1\%$ ($\varepsilon_1 = -1.35$).

Fig. 7 shows the TMA curve of the sample previously deformed with $\varepsilon_1 = -1.35$. The aspect of the curve is very similar to that presented by the AISI 304 stainless steel. A detailed interpretation of this behavior is presented in [10]. The TMA curve can be divided into 3 regions. In regions I and II the curve is reversible and the decrease of magnetization is due to the effect of temperature on the intrinsic magnetization of phase α'. The $\alpha' \to \gamma$ transformation occurs in the region III. The A_s temperature determined as the intersection of regions II and III is 390 ± 10°C. The A_f temperature obtained in the end of region III is 540 ± 10°C. The heating rate used in the TMA of this alloy must be relatively high

(10°C/min) in order to avoid ferrite precipitation by diffusion mechanism.

4. Conclusions

Martensites α' and ε are produced by cold deformation of the Fe-17Mn-1.9Al-0.1 alloy. These two phases were detected by X-ray diffraction in samples deformed with true strains (ε_1) varying from -0.32 to -2.96. The sequence of transformation $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ observed in the common stainless steels is not observed in this material.

The martensite ε is reverted between 300°C and 450°C. Martensite α' is reverted by continuous heating between 390°C (A_s) and 540°C (A_f). When the steel is heat treated between 450°C and 750°C the ferrite phase (α) is formed by diffusion mechanism.

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References

1. J. CHARLES, A. BERGHEZAN and A. LUTTS, *J. de Physique-Colloque* **45**(C1) (1984) 619.
2. C. J. ALTSTETTER, A. P. BENTLEY, J. W. FOURIE and A. N. KIRKBRIDE, *Mat. Sci. and Engineering* **82** (1986) 13.
3. F. C. CHEN, P. LI, S. L. CHU and P. CHOU, *Scripta Metall. et Mat.* **25** (1991) 585.
4. J. C. BENZ and H. W. LEAVENWONTH JR., *J. of Metals* **37** (3) (1985) 36.
5. F. C. CHEN, P. LI, S. L. CHU and P. CHOU, *Mat. Sci. and Engineering* A **160** (1993) 261.
6. P. MANGONON and G. THOMAS, *Metall. Trans.* **1** (1970) 1587.
7. H. MATHIAS, Y. KATZ and S. NADIV, in Proceedings of the 2nd International Congress on Hydrogen in Metals.
8. V. SEETHARAMAN and R. KRISHNAN, *J. Mater. Sci.* **16** (1981) 523.
9. J. Singh, *ibid.* **20** (1985) 3157.
10. S. S. M. TAVARES, D. FRUCHART and S. MIRAGLIA, *J. of Alloys and Compounds* **307** (2000) 311.
11. K. B. GUY, E. P. BUTLER and D. R. WEST, *Metal Science* **17** (1983) 167.
12. J. CHILDRESS, S. H. LIOU and C. L. CHIEU, *J. Appl. Phys.* **64** (10) 6059.
13. "ASM Metals Handbook," Vol.8, 8th ed. (ASM, 1973).

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