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Positron annihilation studies of high-manganese steel deformed by rolling

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Abstract. Positron annihilation spectroscopy (PAS) has been used to study the annealing behavior of cold rolled Fe – 21 wt% Mn steel with 0.05 wt% C. After the initial annealing of defects shown by Doppler broadening of the annihilation line, a slight increase in the annihilation line shape parameter, i.e., the so-called S parameter and then its decrease in the temperature range between 225°C and 450°C indicates generation of new defects and their subsequent annealing. This temperature range coincides with X-ray diffraction measurements, which indicate reversion of deformation-induced ε-martensite. However, for annealing in this temperature range with slow cooling of the sample, the formation of ferrite already starts. The results are compared with our previous results for deformed austenitic stainless steel 1.4301 (EN) where only reversion of deformation-induced α′-martensite was detected.

Key words: plastic deformation • positron annihilation • high manganese steel

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Introduction

High manganese austenitic steels Fe-Mn-C with up to 30 wt% Mn and more than 0.4 wt% C have one of the best combination of the strength and plasticity among the currently available steels. This is due to the twinning-induced plasticity (TWIP) effect that occurs at room temperature but progressively disappears when the deformation temperature increases. Deformation mechanisms also include dislocation glide and ε (hcp)/α′ (bcc) martensite formation. Boundaries of fine twins and the martensite plates/needles formed during plastic deformation act as strong obstacles to dislocation glide that leads to the so-called dynamic Hall-Petch effect [1]. Deformation mechanism depends on the stacking fault energy (SFE) value, which is defined by the temperature and composition of the steel. For the Fe – 22% Mn steel, the SFE is ca. 30 mJ/m^2 at the room temperature [2]. According to the literature, the SFE in the range of 20–30 mJ/m^2 results in stable fully austenitic microstructure with TWIP properties [2–6]. Frommeyer *et al.* [7] indicated the twinning effect in the stable austenite for SFE larger than ca. 25 mJ/m^2. According to Dumay *et al.* [8], for the SFE below 18 mJ/m^2, twinning tends to be replaced by ε-martensite. Wang *et al.* [9] have remarked that a sharp transition from twinning to strain-induced ε-martensite can be induced by a very small difference in the SFE of the order of 5–10 mJ/m^2. However, microstructure of the Fe-Mn alloys strongly depends on the thermal history. In the thermodynamic equilibrium state at the room temperature, the alloys

Fe – 10–29 wt% Mn should have some amount of ferrite [10] and completely austenitic structure requires more than 29 wt% Mn [11].

The positron annihilation techniques are well-known experimental methods used for studies of defects in crystalline matter. After implantation into a sample, positrons localize in regions of lower electron density than in a perfect crystal lattice, i.e., open volume defects and then annihilate with electrons. The locally reduced electron density of the defect causes decrease in the positron annihilation rate and increase in its lifetime. The Doppler broadening (DB) of the annihilation radiation reflects information on the local electron momentum distribution in the sites where the annihilation takes place. Crystal lattice defects generated during plastic deformation such as vacancies and their agglomerates and, to some extent, dislocations can be identified and investigated using positron annihilation techniques.

In our previous publication, we reported the positron annihilation studies of annealing behavior of deformed austenitic stainless steel (SS) 1.4301 (EN) [12]. The annealing of defects induced by plastic deformation and generation of other defects by reverse transformation of deformation-induced martensite (DIM) was observed. In the case of 1.4301 SS for samples with 0.19 and 0.25 volume fractions of deformation-induced α′-martensite, generation of new lattice defects trapping positrons was clearly visible on annealing in the temperature range between 450°C and 650°C. This temperature range coincides with the temperature range of α′-martensite reversion, and it was confirmed by the reduction of magnetization.

The aim of the present investigations is application of DB of annihilation line technique to study annealing behavior of Fe-Mn steel. In that case, mainly ε-martensite is formed during cold deformation. The stability of the martensite phases and the generation of lattice defects connected to phase transformations are of our interests. The X-ray diffraction is used as a complementary method to positron annihilation measurements.

Experimental details

The material under investigation was an Fe-Mn alloy. The composition of the electric induction melted alloy is given in Table 1. The ingot was homogenized at 1200°C for 4 h and hot forged at the same temperature to a forging reduction ratio ca. 1.75. After forging, the ingot was quenched in water. The ingot was cut using water jet technology to obtain slabs of dimensions 160 × 15 × 5 mm^3. The surface of the slabs was polished and two slabs were cold rolled at room temperature to the thickness reduction of 30% (sample A) and 40% (sample B). For positron annihilation measurements, two samples of the length of 10 mm were cut from each slab after cold rolling using low-speed diamond saw. The samples were annealed in steps for 1 h in the flow of N_2 gas in constant annealing temperatures covered the range from room temperature to 800°C. After each annealing, the samples were cooled down with the furnace and the positron measurement was performed using a DB spectrometer with a coaxial high-purity germanium (HPGe) detector, which had the energy resolution equal to 1.4 keV – the full width at half maximum (FWHM) interpolated at 511 keV. The ^{22}Na isotope enveloped in a 7-μm-thick kapton foil was used as positron source. Additionally, the X-ray diffraction measurements were carried out using Cu$K\alpha$ radiation on a Philips X-Pert diffractometer.

Results and discussion

Figure 1 shows X-ray diffraction pattern for the initial sample after forging. The patterns for the sample cold rolled to the thickness reduction $r = 30\%$ and for this sample after annealing at 425°C are shown in Fig. 2. It can be seen that the initial sample exhibits

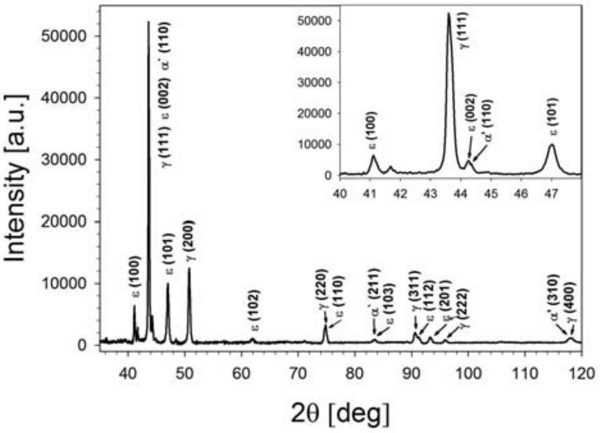

Fig. 1. X-ray pattern of the sample after forging.

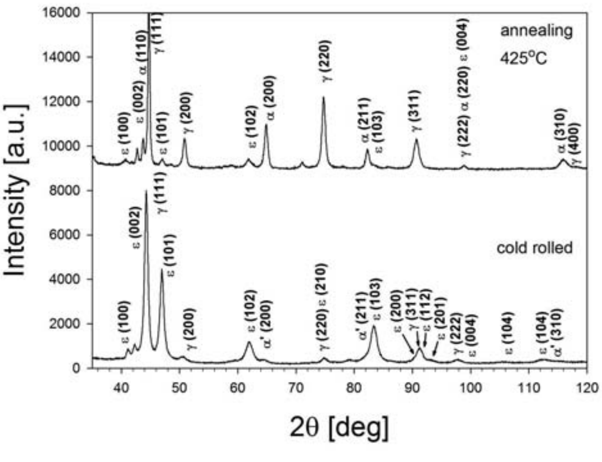

Fig. 2. X-ray patterns of the deformed sample and after annealing at 425°C.

Table 1. The chemical composition of the studied samples [wt%]

Mn	C	Mo	Cr	Si	Cu	Al	Ni	V	S	P	Fe
21	0.05	<0.005	0.1	0.19	0.04	0.006	0.02	0.008	0.003	0.02	Balance

mainly austenite (γ-phase) and some contribution of phases with the hcp structure (ε-martensite) and the bcc one (α'-phase). According to Fig. 6 in Ref. [13], ε-martensite can be produced during quenching of the alloy to the room temperature. In the plastically deformed material, peaks of ε- and α'-martensites are relatively strong. Thus the deformation induced the formation of ε- and α'-martensites at the expense of austenite. On the other hand, after annealing at 425°C, ε-martensite peaks are lowered and the ferrite (α-phase) peaks are significantly increased. Thus, the reversion of ε-martensite (i.e., $\varepsilon \rightarrow \gamma$ transformation) takes place during the annealing treatment at temperatures up to 425°C and the ferrite phase can be formed according to the equilibrium phase diagram Fe-Mn [10]. After cooling the samples to the room temperature, two-phase austenite-ferrite microstructure with very small amount of the ε-martensite was obtained. Formation of ferrite was reported for Fe – 17 wt% Mn – 1.9 wt% Al – 0.1 wt% C alloy, which has also low concentration of carbon. After annealing in higher temperature range, i.e., 450–750°C, large amount of ferrite is formed [14]. The origin of a few small peaks that are not indexed can be iron or manganese oxides. The inclusions of oxides were reported [15].

Figure 3 presents the results of DB measurements of isochronal cumulative annealing of the cold rolled Fe-Mn alloy samples. The temperature dependence of the S parameter, which is defined as the ratio of the area under the fixed central part of the annihilation line to the area under the whole annihilation line, is depicted in the figure. The S parameter is sensitive to the annihilation of positrons with low momentum electrons that are present in open volume defects.

Four stages in the annealing curve can be distinguished. Stage I between 100°C and 225°C is characterized by the decrease in the S parameter. The value of the S parameter after cold rolling is higher for the sample with higher degree of deformation. The decrease indicates annealing of defects induced by deformation. After this stage, the S parameter values for the samples studied do not differ significantly. A similar behavior was observed for deformed

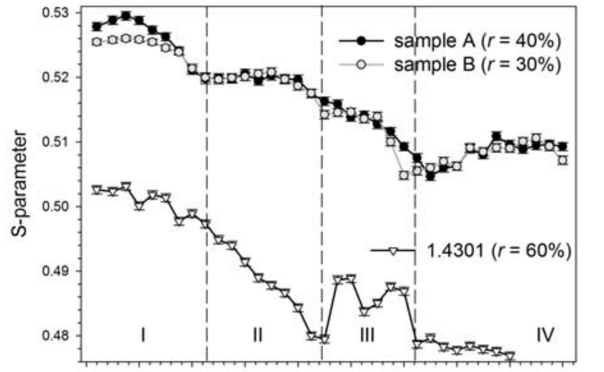

Fig. 3. The S parameter dependence on annealing temperature for two cold rolled samples of Fe – 21.6 wt% Mn – 0.7 wt% C steel and cold rolled austenitic SS 1.4301 (EN). The thickness reductions are given in parentheses.

1.4301 SS and 1.4307 SS [12, 16, 17], but in that case, the decrease starts at higher temperature of about 200°C and continues up to 450°C, open triangles in Fig. 3. For the studied Fe-Mn alloy samples, the dependence of the S parameter on temperature is quite different. The S parameter slightly increases in stage II and starts decreasing again at 375°C for the sample A. It seems that the temperature at which the decrease starts for the sample B is slightly shifted to 425°C. This behavior indicates generation of defects and their subsequent annealing that can be caused by the reversion of ε-martensite. This is in agreement with the X-ray diffraction results. However, the presence of ferrite grains can also contribute some defects connected to, e.g., grain boundaries. It seems that the temperature range of ε-martensite reversion depends slightly on the degree of deformation. A temperature range similar to that of ε-martensite reversion, i.e., 300–450°C was found for Fe – 17 wt% Mn – 1.9 wt% Al – 0.1 wt% C alloy [14]. In both cases, the temperature range is higher than that usually observed in SSs [18]. Lü *et al.* determined the start and finish temperatures of reverse transformation of ε-martensite to austenite for Fe – 21.6 wt% Mn – 0.38 wt% C alloy [19]. The temperature range obtained by them is 200–375°C. They also reported that the austenite that transformed from ε-martensite was highly dislocated. Dislocations yielded by the reverse transformation $\varepsilon \rightarrow \gamma$ are able to move easily and react with other dislocation creating point defects, e.g. dislocation jogs or vacancies that are deeper traps for positrons. This process can contribute to the increase in the S parameter. Subsequently, vacancies can migrate to grain boundaries, which constitute their sinks. Annealing of defects should cause decrease in the S parameter. For 1.4301 SS, ε-martensite is not present and there is no ε-martensite reversion. Hence, only the decrease in the S parameter in this stage is observed, which indicates the decrease in concentration of positron trapping defects induced by plastic deformation, that is, vacancy-type defects associated with dislocations.

Within stage III, the S parameter for the Fe-Mn alloy initially is almost constant and then decreases, which suggests further annealing of defects. The defect generation is less pronounced than during the ε-martensite reversion in stage II. The temperature range of stage III coincides with the increase in the S parameter for the deformed 1.4301 SS, which was ascribed to the generation of defects during α'-martensite reversion [12]. Volume contraction accompanying bcc or fcc change during α'-martensite reverse transformation results in volume excess that may contribute to the vacancy cluster formation. However, distinct increase in the S parameter for the Fe-Mn alloy in not pronounced as it takes place for 1.4301 SS. In the case of the Fe-Mn alloy studied, it may be caused by ferrite formation. The decrease in the S parameter at temperatures higher than 550°C can also be caused by recrystallization. Lü *et al.* studied recrystallization kinetics in Fe – 21.6 wt% Mn – 0.38 wt% C alloy. They found that for the sample cold rolled to the thickness reduction of 50% annealing at 560°C for 3 h resulted in a recrystallized volume fraction of about 80% [20]. The S parameter has the minimum value at temperatures 600°C for sample A and 650°C for sample B. The small increase in the S parameter at higher temperatures (stage IV) indicates that the defect concentration slightly increases, which can be related to the fact that the alloy is not monophase and boundaries between grains of different phases can be positron trapping sites.

Conclusions

The PAS studies were carried out on cold rolled Fe-Mn alloy to investigate the microstructural changes as a function of cumulative isochronal annealing. The temperature dependence of the annihilation line shape parameter, i.e. the S parameter, shows four stages. In stage II, the reversion of the deformation-induced ε-martensite is reflected in the slight increase and subsequent decrease in the S parameter in the temperature range 225–450°C. This is attributed to generation and annealing of defects created in this process. For higher annealing temperatures, a number of processes such as reversion of deformation-induced α′-martensite, ferrite formation during slow cooling and recrystallization can take place. To distinguish the influence of these processes on the positron annihilation characteristics, further researches are needed.

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