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Physical, microstructural and mechanical study of isochronal annealing of deformed commercial iron

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ABSTRACT

Isochronal annealing of deformed commercial iron samples (99.88 m.-% Fe) after cold deformation is studied by means of Vickers microhardness and physical techniques based on positron annihilation (positron annihilation lifetime spectroscopy (PALS) and Doppler broadening of the annihilation radiation (DBAR)). Undeformed Fe with a purity of 99.998 m.-% was used as a reference material to compare it with the used commercial Fe. Additionally, EBSD (electron backscatterded diffraction) was used to estimate the recrystallization fraction in the samples. In the temperature range 600–1000 °C the recrystallization phenomenon can be followed with these techniques and similar results are obtained, while in the temperature range 20–600 °C the recovery process has to be included and the results for positron annihilation lifetime presented a deviation due to its sensitiveness for changes in point defect configurations.

1. Introduction

Different techniques can be used to study the evolution of plastically deformed iron during its isochronal annealing: mechanical testing (Vickers hardness), microstructural examination (metallography, electron backscattered diffraction (EBSD),…) and physical techniques involving positron annihilation (PA), such as PALS and Doppler broadening of the annihilation radiation (DBAR).

Elements of the microstructure were determined by electron backscattered diffraction (EBSD) in a scanning electron microscope (SEM). This technique provides information on the crystallographic orientation of each grain [1].

Although PA techniques are well established in the area of experimental physics, in the industry and the more conventional research centers PA-techniques are not extensively used. There is a need to better correlate the conventional techniques based on mechanical testing and microstructural examination with the fundamental physical technique of the PALS and the DBAR.

PALS can quantify the size of open volume defects, as well as the defect concentration and is based on the precise measurement of the lifetime of a positron in a solid. The defect concentration is calculated from the different fractions of positrons that annihilate in the traps. The defect size and type is directly related to the value of the positron lifetime: the larger the defect, the lower the local electron density and consequently the longer the positron lifetime will be [2].

Values of the lifetime of positrons trapped in dislocations are close to or slightly below those for the vacancy lifetime [3–5]. For this reason, it is normally accepted that the positron lifetime can be related to vacancies trapped in the stress field around a dislocation line or in vacancies on a dislocation line, which would be equivalent to a pair of mono-atomic jogs [6]. Hidalgo et al. [7] measured the positron lifetime in deformed iron to be 150 ps. They suggested that positrons annihilate at associated defects (vacancies or dislocation jogs) rather than at the dislocation line. Park et al. [8] have studied the effectiveness of positron trapping by edge and screw dislocations and made an attempt to determine the fraction of dislocations of each type per unit area, although the difference in lifetimes is rather small. Calculation of positron lifetimes in jogs and vacancies

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on an edge dislocation line in Fe was also reported by Yasushi Kamimura et al. [9].

The Doppler Broadening of the Annihilation Radiation (DBAR) is based on the measurement of the linear momentum of the annihilating electron-positron pair. The photons created during the electron-positron annihilation are detected by a germanium detector. The shape of the resulting photo-peak reflects the momentum distribution of the original electron-positron pair. This distribution is determined by the momentum distribution of the electrons seen by the positron, which is influenced by the size and the nature of the defects. Measurements of the Doppler broadening of the positron-electron annihilation radiation are generally characterized by the S (Shape) parameter, defined as the ratio between the surface of the central part of the annihilation spectrum and the total surface of the spectrum. This parameter reflects the positron annihilation with valence electrons (low momentum). In general, a high value of S indicates positron annihilation in open volume defects (vacancies and vacancy groups). A second useful parameter for the analysis of the DBAR is the W (Wing) parameter, which reflects the positron annihilation with high momentum electrons (core electrons). It is defined as the ratio of the counts in both side wings of the spectrum over the total number of counts in the annihilation line.

The plastic deformation of metals and alloys provokes changes in the density and distribution of different kind of structural defects (mainly vacancies and dislocations), whereas the positrons are captured by dislocations and vacancies and their lifetimes increase. In polycrystalline samples, the deformation phenomena are relatively complex due to various interactions between dislocations and grain boundaries [4–6]. It is well known that cold deformation of metals such as a rolling process induces a high amount of defects (mono, di-vacancies, vacancy-interstitial agglomerates, dislocations,…) [10]. Plastic deformation of metals occurs by the generation and movement of dislocations, which stores a certain amount of deformation energy along each dislocation line in the form of an elastically distorted region. During deformation the dislocations gather together and produce tangled groups distributed over local bands and forming tangled networks defining 'cells' with a lower concentration of dislocations and a slight orientation difference [11–13].

The microstructural changes during the annealing of deformed metals are described in terms of recovery, recrystallization and grain growth (with increasing duration and temperature of the annealing process). Different rearrangements of the dislocation distribution occur during the annealing, depending on the amount of deformation. In the case of small deformation strains the annealing causes the dislocations to rearrange into local bands, similar to grain boundaries and may develop into a sub-grain structure. In metals deformed with large deformation strains a softening prior to the onset of recrystallization appears to be controlled by sub-grain growth [14].

Very pure iron will recrystallize easily (short time, lower temperature), whilst the presence of impurities (even in small concentrations) will clearly increase the resistance to recovery and recrystallization. Interstitial elements have an important effect on the recovery processes, even when present in very low concentrations: they are considered to be responsible for the retardation of recovery [15,16]. Ohkubo et al. stated that dislocations and vacancy clusters are introduced in bcc Fe regardless of the type or degree of deformation [17]. Hamdy F.M et al. studied cold-worked iron with different percentages of deformation up to 40% using the PALS [18]. Vacancy clusters, as well as dislocations are produced as a result of a cold-working. In our work only dislocation are detected as a result of the high degree of deformation (75% thickness reduction) and also the different kind of deformation.

2. Experimental procedure

The effect of annealing of the commercial iron samples with a purity of 99.88 m.-% is studied using the PALS. The samples were cold rolled at room temperature with a thickness reduction of 75%. A totally annealed high purity Fe sample (with a purity of 99.998 m.-%) was used as a defect free sample for the reference. The chemical composition of the commercial iron samples used in the present work was determined with a LAVWA18A spectrometer of Spectro Analytical Instruments and is shown in Table 1.

After cold deformation the samples were annealed at temperatures from 100 °C (boiling distilled water) to 1000 °C (vacuum furnace) during a constant time of one hour (isochronal annealing) in temperature intervals of 100 °C. After annealing, each sample was cooled down in the furnace until room temperature.

The evolution of the microstructure and corresponding properties during the process of isochronal annealing were investigated using different techniques: mechanical testing using the Vickers micro-hardness, the characteristics of the metallographic microstructure were observed by the EBSD and finally two physical techniques based on positron annihilation were used: PALS and DBAR.

Vickers microhardness (H_V) tests applying 300 g were performed on the surface of deformed and annealed commercial iron samples to quantify the softening fraction; at least six indentations were done in each material.

The microstructure of the samples was evaluated after polishing with a colloidal silica solution by means of optical microscopy and EBSD scans. The EBSD-measurements were carried out on an FEG SEM Zeiss Ultra Plus microscope equipped with an hkl EBSD system and maps were generated with a step size of 1 μm, covering a total area of 400 × 400 μm^2.

Positron lifetime measurements were performed at room temperature using a fast–fast lifetime spectrometer [19]. Each spectrum contained more than 10^6 counts and several spectra were accumulated for each sample in order to ensure the reproducibility of the data. The Doppler broadening (DB) of the 511 keV annihilation line was measured and the results were analyzed in terms of the so-called S and W parameters. The DBAR and the positron lifetime measurements were performed at room temperature after each annealing process.

3. Results

3.1. Softening behavior studied by microhardness measurements

Vickers microhardness (H_V) measurements for isochronally annealed samples are shown in Fig. 1. The error bar shows that there is a considerable scatter on the measurements. The microhardness of the cold rolled sample without any annealing (H_V^{CR}) is 209, whilst the hardness of the fully recrystallized material (H_V^{FR}) is 80, (the sample annealed at 700 °C is considered to be completely recrystallized). In the annealing temperature range 100–500 °C there is a slight reduction of hardness, which can be attributed to a recovery process. The hardness drops very clearly between 500 and 700 °C because of the recrystallization of the material. Above 700 °C the hardness values further decrease slightly due to grain growth as the annealing temperature increases.

A softening fraction R for the isochronal annealed samples can be defined as follows:

Table 1
Determined impurities in the used commercial iron sample [in ppm].

C	Si	Al	Nb	Cr	Mo	Ni
20	80	560	300	50	50	50