Hyperfine Interact (2016) 237:45
DOI 10.1007/s10751-016-1228-2

Study of the changes in the magnetic properties of stainless steels under mechanical treatment

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Abstract Six types of stainless steels (SS) were studied for changes in its structure and magnetic properties under mechanical treatment. Depending on intensity and duration of the process of plastic deformation and the SS type the paramagnetic austenite structure transforms partially to completely into ferrite structure with ferromagnetic behaviour. Some of the SS tested were found slightly modified yet in the process of its manufacturing. Only one SS type with high Ni content preserved its structure and paramagnetic properties even after very intense mechanical treatment.

Keywords Mössbauer spectroscopy · Stainless steel · Mechanical treatment · Magnetic properties

1 Introduction

For the construction of a large volume (about 1 m^3) vacuum chamber for the so called "iron free orange" β-spectrometer [1] different stainless steel (SS) types are tested by Mössbauer spectroscopy, magnetic susceptibility measurements and other methods. Such

This article is part of the Topical Collection on *Proceedings of the International Conference on the Applications of the Mössbauer Effect (ICAME 2015), Hamburg, Germany, 13–18 September 2015*

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Published online: 22 February 2016

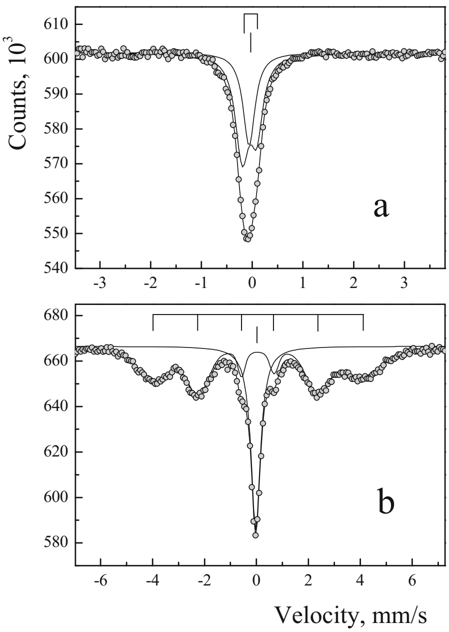

Fig. 1 Mössbauer spectra of SS type 1X9H10T taken at room temperature: **a** sample without mechanical treatment at velocity range about ±4 mm/s; **b** sample after 40 min mechanical treatment performed in a one-ball vibration mill at velocity range about ±7 mm/s. A new component with Zeeman-split subspectrum and ferromagnetic properties appears

studies are not new [2–4]. In our case it was important to check whether or not, after plastic deformations like stretching, bending, forging etc. the construction material preserves its paramagnetic properties. Ferromagnetic behavior is unfavorable because it could produce changes in the toroidal configuration of the magnetic field within the vacuum chamber which is thought to focus the β-particles. In this contribution we describe the effect of the intensity and duration of the process of plastic deformation on different types of stainless steel and cases of partial to complete transformation from austenite to ferrite structure.

2 Experimental

This study started with the investigation of the four types 1X9H10T, 1X18H10T and analogs SS plates with 10 mm thickness. Further two SS types 12X18H10T and 10X23H18 have been chosen for more precise and detailed studies. The first SS type was chosen for its broad applications from commodity such as scissors or cutlery to construction material for some reactor vessels in chemical industry. The second type was chosen for its high Ni concentration. The sampling was done by diamond filing from the bulk objects. The Mössbauer spectroscopy studies have been performed in transmission geometry at room temperature with a standard spectrometer working in constant acceleration mode. Mössbauer sources ^{57}Co in Rh matrix and activity of about 25 mCi were used. The spectra were processed with the software packets VINDA and CONFIT 2000. The first mechanical treatments were performed in a small one-ball (agate, diameter 60 mm) vibration mill with different duration: 20, 40 min, 1 and 2 h. The mill didn't provide intense and reproducible mechanical activa-

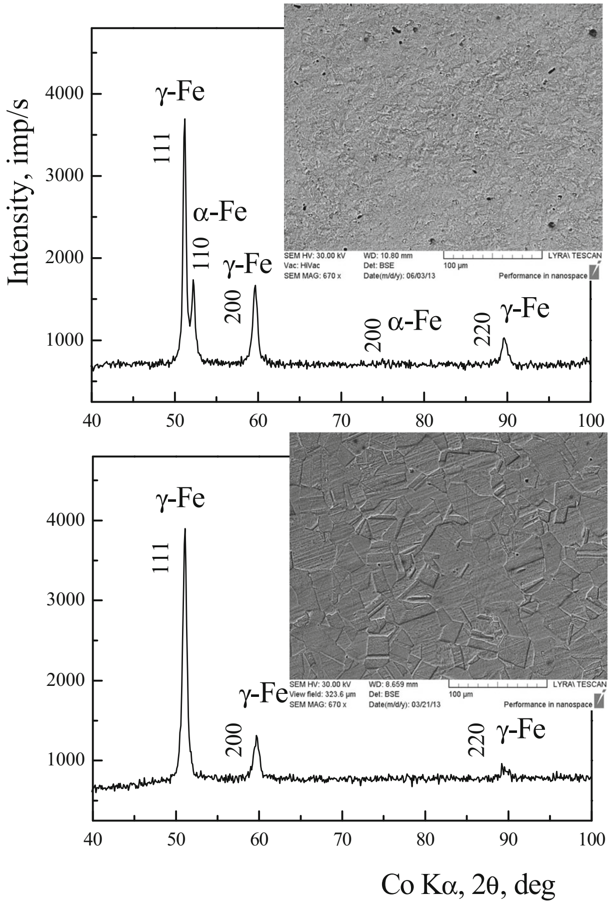

Fig. 2 X-ray diffraction studies: (*top*) SS type 12X18H9T and scanning electron microscopy image; (*bottom*) the same studies for SS type 10X23H18

tion, so for the second set we used a planetary driven Retsch PM100 type mill. The X-ray energy dispersive analysis (EDAX) and images in back scattered and secondary electrons have been obtained by a scanning electron microscope TESCAN, LYRA/FIB-SEM. The microelements concentration was measured by ICP-MS Perkin Elmer DRC analyzer. The crystal structure studies have been performed on a TUR-M62 diffractometer. The magnetic susceptibility measurements have been done on a self-made vibrational magnetometer.

3 Results and discussions

The Mössbauer measurements of samples without mechanical treatment, Fig. 1 a exhibit only an austenite single Mössbauer line with complicated profile centered at nearly zero velocity. Old studies pointed out the deviation of the experimental line profile from lorentzian one [5, 6]. The first alloying neighbors are Cr and Ni. They change slightly the isomer shift and produce unresolved quadrupole doublets or a doublet with a small quadrupole splitting. Detailed study and approximations with singlet (pure γ-Fe structure)

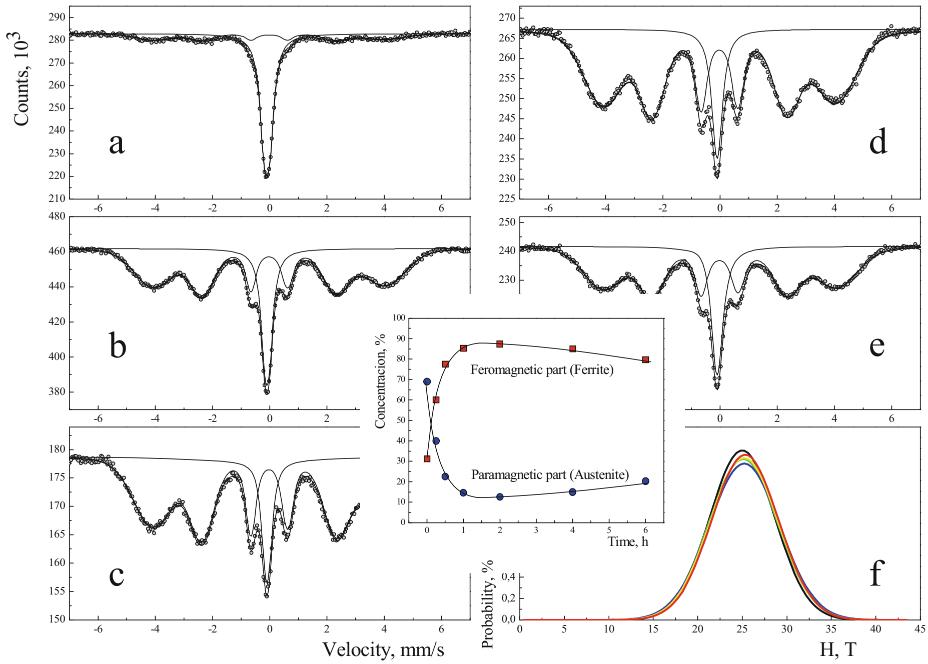

Fig. 3 Mössbauer spectra of SS type 12X18H9T taken at room temperature: **a** without mechanical treatment; After mechanical treatment: **b** 30 min; **c** 1 h; **d** 2 h and **e** 6 h. **f** the normalized magnetic hyperfine field distribution extracted from the spectra. The dynamics of the transformation from austenite into ferrite is given in the middle

and up to five quadrupole doublets (different first nearest neighbors configurations in the first coordination sphere) are described [7]. A good approximation of the experimental line profile (only for measurements in velocity range about ± 4 mm/s) can be achieved by fitting the experimental spectra to one singlet and one symmetric quadrupole doublet. We have chosen to fit the spectrum to one singlet and one asymmetric doublet. The asymmetric doublet accounts better for the summative influence of the several (up to five) alloying neighbors, Fig. 1a. Relative short in duration (20 ÷ 40 min) and not very intensive mechanical treatment, Fig. 1b caused substantial transformation of austenite into ferrite detected as a second subspectrum with a broad magnetic hyperfine field distribution. The distribution is normally centred at about 25 T and the magnetic properties are changed from para- to ferromagnetic. In some cases again to achieve a better approximation of the experimental spectrum a second distribution centered at about 33 T was added, which probably is related to an iron-rich component [3]. One to two hours of mechanical treatment in the mill are enough for complete transformation (more than 90 %) of the sample from austenite to ferrite. One of four SS types without mechanical treatment exhibits not only an austenite singlet but also broad sextet with distribution which shows that the material is slightly modified yet in the process of its manufacturing. Such transformations are strongly unfavorable and duraluminium plates were used for the construction.

Further two SS types 12X18H10T and 10X23H18 have been chosen for more precise and detailed studies. The standard concentration of the main alloying elements has been confirmed by X-ray fluorescence analysis and the microelements concentration has been confirmed also by ICP-MS analysis. The performed X-ray diffraction studies for

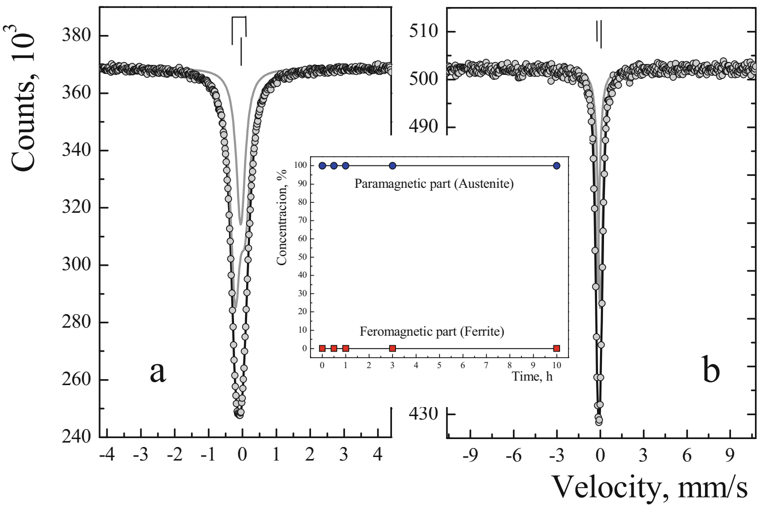

Fig. 4 Mössbauer spectra of SS type 10X23H18 taken at room temperature: **a** sample without mechanical treatment at velocity range ±4 mm/s; **b** sample after one hour mechanical treatment taken at larger velocity range. Only this stainless steel with high Ni content preserves its structure and paramagnetic properties unchanged even after very intense mechanical treatment (inset)

SS type 12X18H9T show face centered cubic fcc lattice (γ-Fe) partially modified to body centered cubic bcc lattice (α-Fe) which is not typical for austenite structure, Fig. 2 (top). Scanning electron microscopy image shows very fine crystallites again not typical for austenite microstructure, Fig. 2 (top inset). This is not exactly SS because the material is slightly modified yet in the process of its manufacturing. The second SS type 10X23H18 shows only fcc lattice and large twin blocks typical for SS austenite structure, Fig. 2 (bottom).

In this second set of measurements both SS types are mechanically activated equal in intensity and duration by using a planetary driven type mill. Mössbauer spectra of SS type 12X18H9T taken at room temperature without mechanical treatment as well as X-ray diffraction studies show, Fig. 3a that the material is slightly modified. After mechanical treatment of about 1.5 h maximal transformation up to 90 % has been measured. Unexpected back bending effect and lowering of the transformation to about 80 % has been observed after 6 h mechanical treatment. The dynamics of the transformation from austenite into ferrite is given in the middle on Fig. 3. The normalized magnetic hyperfine field distributions extracted from the spectra are nearly the same, Fig. 3f.

Mössbauer spectra of SS type 10X23H18 were taken at room temperature without mechanical treatment and after one hour mechanical treatment are shown on Fig. 4a and b. It was confirmed that only this SS type with high Ni content preserves its structure and paramagnetic properties unchanged even after very intense mechanical treatment, Fig. 4 (inset).

Five out of the six SS types tested have undergone austenite to ferrite transformation after plastic deformation. Mössbauer spectra, Fig. 5a, and magnetic susceptibility measurements, Fig. 5a (inset) recorded at RT show that the sample without mechanical treatment exhibits an austenite single Mössbauer line with complicated profile and paramagnetic properties. After 40 minutes of mechanical treatment Zeeman-split magnetic subspectrum with broad

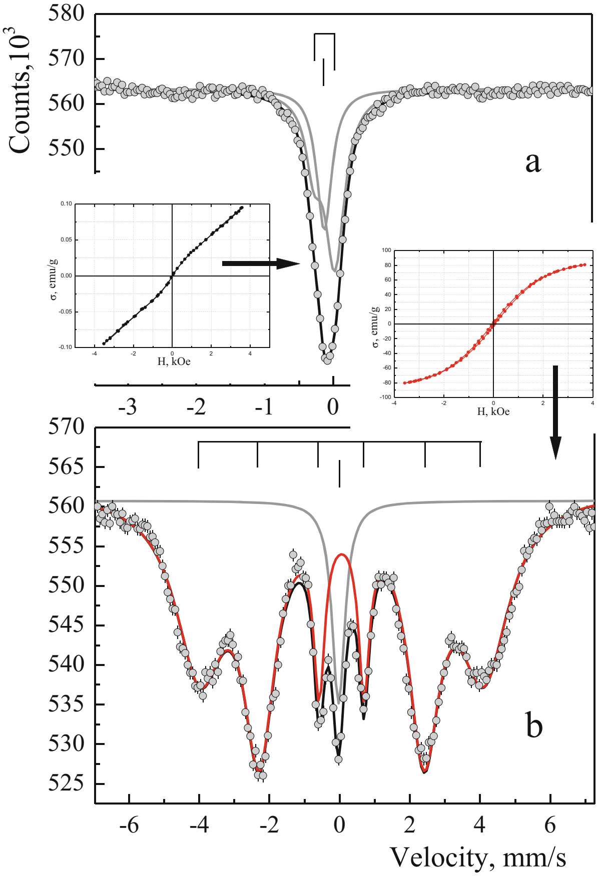

Fig. 5 Example of austenite to ferrite transformation after plastic deformation: Mössbauer spectrum and magnetic susceptibility (inset) recorded at RT. **a** Sample without mechanical treatment exhibits an austenite single Mössbauer line with complicated profile and paramagnetic properties (inset). **b** After 40 minutes of mechanical treatment a Zeeman-split magnetic subspectrum with broad distribution of the hyperfine field is observed in addition to a single line. The property of the main component is ferromagnetic (inset)

distribution of the hyperfine field is observed in addition to a single line, Fig. 5b. Magnetic susceptibility measurements confirmed the ferromagnetic property of the main component, Fig. 5b (inset).

4 Conclusions

The major conclusions of this study are: 1) Even low intense plastic deformation could strongly change the austenite structure and hence its paramagnetic properties. 2) If the deformation of bars, plates, pipes etc. occurs in the cold forming process subsequent thermal annealing is needed to restore the original austenitic stainless steel structure. 3) In Cr-Ni stainless steels with low Ni content mechanical treatment can cause complete transformation from para- to ferromagnetic behavior. 4) However, in stainless steels with high Ni content the structure and paramagnetic properties remain unchanged even after very intense mechanical treatment [8].

As a result of the above studies for the purpose of constructing a large volume vacuum chamber we have proposed to use duraluminium plates.

Acknowledgments V. Rusanov and A. X. Trautwein acknowledges the continuous support by the Alexander von Humboldt-Foundation. Thanks are due to Prof. V. Anchev for helpful discussions. The partial financial support of the Research Fund of the University of Sofia (Contract No. 150/2015) is highly appreciated.

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