

Mössbauer study of deformation induced martensitic phase transformation in duplex steel

Artur Blachowski,
Krzysztof Ruebenbauer,
Jerzy Jura,
Jan T. Bonarski,
Thierry Baudin,
Richard Penelle

Abstract Samples of cold rolled UR45N austenitic-ferritic duplex steel with cold rolling reduction ranging from 0% to 80% were investigated in the form of foils and powders by means of Mössbauer transmission spectroscopy. It was found that cold rolling has a minor effect on the martensitic transformation even at high reductions in the material under investigation. On the other hand, powdering process induces strong martensitic phase transformation, i.e., about 25% of austenite transforms into martensite during powdering. High rolling reductions as well as powdering reduce an average hyperfine magnetic field at iron by about 1 T, while annealing at about 430°C increases this field by about 3 T.

Key words duplex steel • martensitic transformation • Mössbauer spectroscopy • plastic deformation

Introduction

Steels of the austenitic-ferritic duplex structure are characterized by high mechanical strength and good corrosion resistance. They are widely used in chemical, petrochemical and shipyard industries. One of the possible mechanisms of the plastic deformation in duplex steels is strain induced martensitic transformation leading to the transition of paramagnetic austenite into ferromagnetic martensite. Austenite has fcc crystal structure, while martensite at low carbon concentration crystallizes in bcc structure. Transformation efficiency and the amount of austenite transformed into martensite depends upon chemical composition, temperature, strain rate and degree of deformation. Transformation of austenite into martensite changes mechanical properties of the steel leading to its strengthening. Reick [5] found, that in duplex steels having the following chemical composition:

Fe-24.16Cr-5.48Ni-3.27Mo-1.20Mn-0.54Si-0.153N-
-0.022C-0.021P-0.001S wt.%

30% of austenite transforms into martensite at 60% of cold rolling reduction. Some alloying components are inhibitors of this transformation. It is well known that manganese is such an inhibitor [3]. It is not easy to identify martensite in the cold rolled steel analyzing diffraction patterns obtained in transmission electron microscopy.

Material and methods

Hot rolled sheet of duplex steel UR45N having chemical composition, shown in Table 1, was examined. In order to look upon strain induced martensitic transformation, the samples were cold rolled up to 40, 60 and 80% reduction. Figure 1 shows microstructure of the undeformed sample, and the samples reduced to 40% and 80%, obtained by

A. Blachowski✉, K. Ruebenbauer
Mössbauer Spectroscopy Division,
Institute of Physics, Pedagogical University,
2 Podchorążych Str., 30-084 Kraków, Poland,
Tel.: +48 12/ 662 63 17, Fax: +48 12/ 637 22 43,
e-mail: sfblacho@cyf-kr.edu.pl

J. Jura
Institute of Technology, Pedagogical University,
2 Podchorążych Str., 30-084 Kraków, Poland
and Institute of Metallurgy and Materials Science,
Polish Academy of Sciences,
25 Reymonta Str., 30-059 Kraków, Poland

J. T. Bonarski
Institute of Metallurgy and Materials Science,
Polish Academy of Sciences,
25 Reymonta Str., 30-059 Kraków, Poland

T. Baudin, R. Penelle
Laboratoire de Physico-Chimie de l'Etat Solide,
UMR CNRS 8648,
Université de Paris Sud,
410 Bâtiment, F-91405 Orsay CEDEX, France

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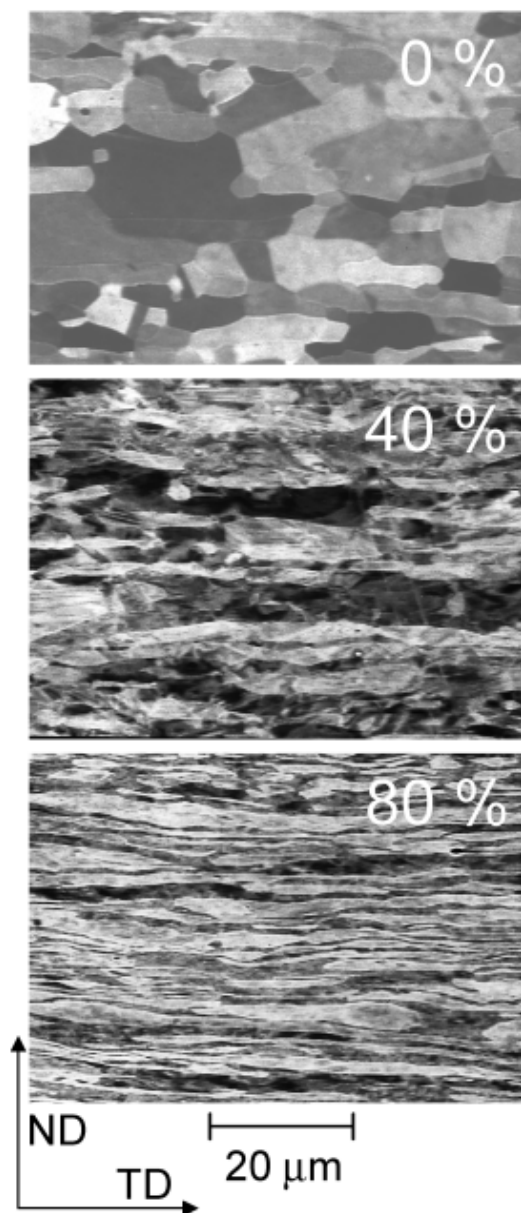


Fig. 1. Microstructure evolution as a function of the cold rolling reduction. ND is a direction perpendicular to the rolling plane, while TD is a direction parallel to the roller axis.

electron scanning microscopy [7]. One can see that grains are strongly deformed and expanded in the rolling plane.

Samples for Mössbauer investigations were prepared as foils having about 100 μm in thickness with a surface parallel to the rolling plane, and as powders obtained by abrasion applying a diamond file. For the latter case absorbers were made by mixing the powder with an epoxy resin and using 30 mg/cm^2 of the steel. Total amount of steel used to prepare a single absorber was about 150 mg. ^{57}Fe Mössbauer absorption spectroscopy was applied to identify phases and to estimate amount of each of them. A single line $^{57}\text{Co}(\text{Rh})$ source of 15 mCi activity was used.

Table 1. Chemical composition of UR45N duplex steel (wt.%).

Cr	Ni	Mo	Mn	N	Cu	C	S
22.54	5.41	3.00	1.86	0.16	0.14	0.019	0.0007

This method is well suited for the purpose as martensite and austenite are easily distinguishable at room temperature because the former is ferromagnetic, while the latter is paramagnetic. On the other hand, absorption spectroscopy requires such an amount of material that averaging over grains is statistically significant. Unfortunately, a distinction between ferrite and martensite is practically impossible by means of Mössbauer spectroscopy.

Contributions of particular phases to the spectra were determined applying transmission integral algorithm. It has been assumed that recoilless fractions are the same in both phases. Subspectrum of the austenite was fitted as unresolved doublet due to spurious electric quadrupole interaction leading to the 0.1 mm/s separation of the spectral components. Subspectrum of the martensite and ferrite is a superposition of many magnetically split components fitted by distribution of the hyperfine effective magnetic field. Distributions of hyperfine interactions are due to multiple local configurations of the alloying components around the iron atom. Spectra for the undeformed and for those having 80% reduction foils are shown in Fig. 2.

For evaluation of the volume fractions of both the (austenite and ferrite/martensite) phases, a new, the so termed incomplete pole figure intensity (IPFI) method might be applied [1]. This direct comparison method is based on the back-reflection pole figures measured by means of X-ray diffraction technique for chosen reflections of both the phases. The experimental data allow to calculate

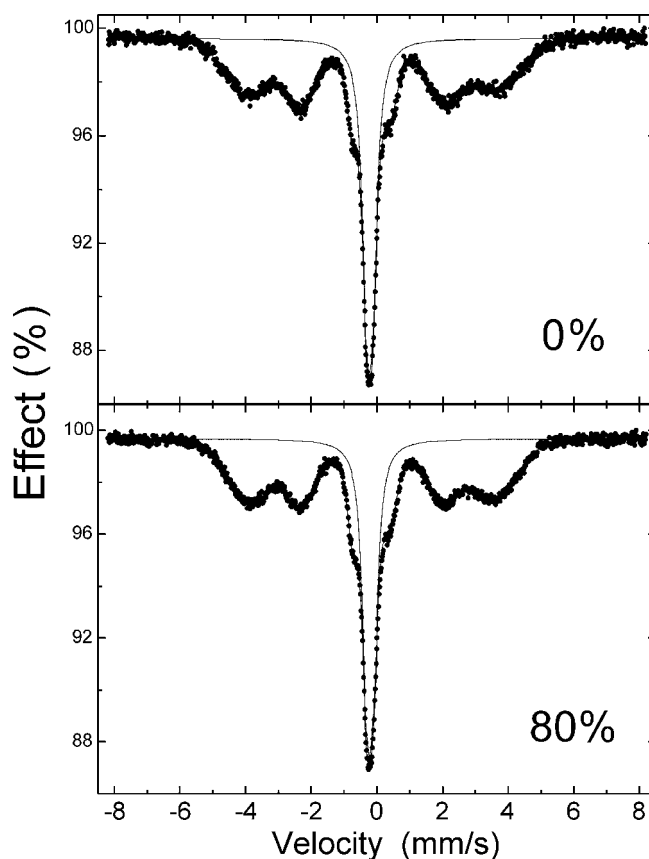


Fig. 2. Room temperature ^{57}Fe Mössbauer spectra recorded on foils undeformed and having 80% rolling reduction. Subspectrum of the austenite has been marked by an additional line.

Table 2. Phase composition and hyperfine magnetic fields. Isomer shift of the austenite depends neither upon the rolling reduction nor powdering, and amounts to -0.10 mm/s vs. room temperature metallic iron. All data were obtained at room temperature.

Rolling reduction (%)	Foil austenite abundance (%)	Powder austenite abundance (%)	Foil martensite/ferrite average magnetic field (T)	Powder martensite/ferrite average magnetic field (T)
0	53	32	23.0	21.9
40	51	30	23.1	22.1
60	51	21	23.0	22.2
80	49	22	22.3	22.1

the so called orientation distribution function for each phase, and hence complete pole figures, the latter being used after integration in calculation formula of the IPFI method.

Electron back-scattered diffraction analysis (EBSD) is another possibility of quantitative analysis of the two-phase steel [4]. The area inside which EBSD measurements are carried out is restricted to a few square millimeters and the information is collected from a thin surface layer. It is very difficult to characterize the microstructure by EBSD measurements when the rolling reduction is too high.

Both of the above methods collect information from relatively small volumes of the sample, and thus resulting austenite and ferrite/martensite contents are not always statistically representative.

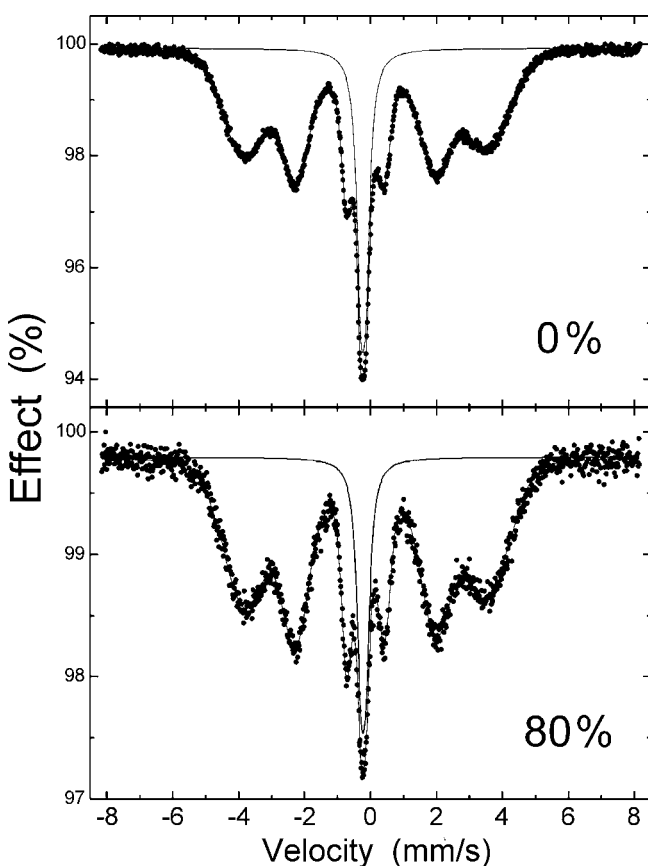


Fig. 3. Room temperature ^{57}Fe Mössbauer spectra recorded on powder obtained from samples undeformed and having 80% rolling reduction. Subspectrum of the austenite has been marked by an additional line.

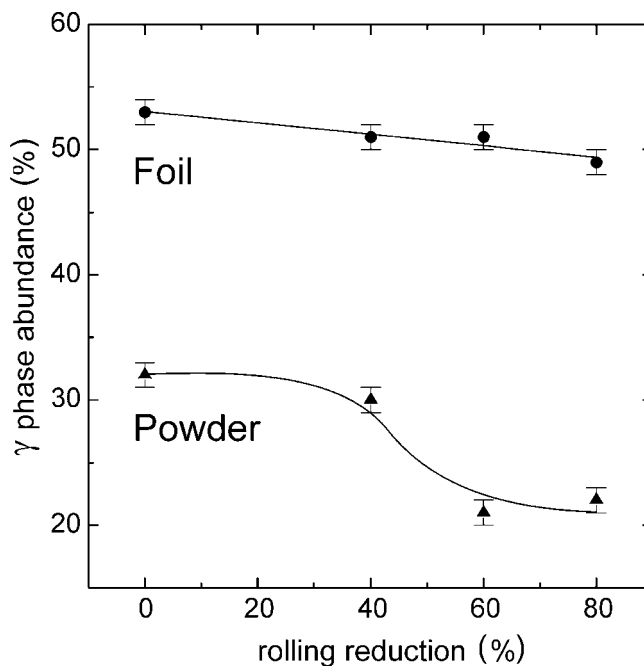


Fig. 4. Abundance of the austenite in foils and powders vs. rolling reduction.

Results and discussion

Essential results obtained by Mössbauer spectroscopy are summarized in Table 2. There is a slight trend showing that martensitic transformation occurs upon cold rolling with approximately a 1% decrease of the austenite content with a 20% increase in rolling reduction. Powder spectra are shown in Fig. 3 for steel samples having the same reduction as foil spectra shown in Fig. 2. Results obtained for powders indicate that powdering significantly stimulates martensitic transformation for the duplex steel UR45N. A difference in the abundance of the austenite between bulk and powder samples is about 25% on the average. Abundances of the austenite in foils and resulting powders vs. rolling reduction are shown in Fig. 4.

An average hyperfine magnetic field is smaller in powders than the field in the corresponding foils. This effect is due to the creation of dislocations during powdering and cold rolling processes. Powdering and high rolling reduction (80%) reduce the field by about 1 T.

Foil having rolling reduction of 80% has been annealed at 430°C for 27 h. The abundance of the austenite has not changed upon annealing despite removal of the residual stress introduced by cold rolling. Hence, one can conclude that stress relaxation due to annealing is insufficient to induce martensitic transformation. The average magnetic field in a foil sample increased from 22.3 T to 26.0 T after annealing, probably due to the removal of extended structure defects, while average fields in powders practically do not depend upon previous thermal and mechanical treatment of the sample prior to powdering. It is well known that Fe-Cr alloys decompose into Fe-rich and Fe-deficient phases at elevated temperatures [2]. However, this process is absent here as the spectra of the powders made from the rolled sample and the sample annealed after rolling have the same hyperfine parameters.

In order to be sure that no material is introduced into the powder from the file, a piece of technical aluminum was powdered in an amount exceeding five times that used to make powder absorbers. A spectrum of such a powder sample was collected and showed no signs of magnetically split components. One has to note that the core of the diamond file is made of the ferritic steel.

Conclusions

Transformation from austenite to martensite leads to the state of the lower energy in the vicinity of the room temperature, as the latter phase is stable, while the former is meta-stable. Plastic deformation energy introduced by cold rolling or powdering of the UR45N steel can stimulate this transition. In the bulk material, the transition is almost inhibited despite introduction of the above energy as the transformation from the austenitic phase to the martensitic phase requires relaxation of the strain. Such a change is suppressed due to the lack of freedom caused by the surrounding matrix. Once the surface has been exposed due to the powdering, the transformation proceeds. A similar transformation due to mechanical stresses was observed by Skrzypek *et al.* [6] in hard ball bearing steels.

Hence, one has to be careful while preparing powder Mössbauer absorbers of some materials, since the phase composition might change during powdering.

References

1. Bonarski JT, Wróbel M, Pawlik K (2000) Quantitative phase analysis of duplex stainless steel using incomplete pole figures. *Mater Sci Technol* 16:657–662
2. Cieślak J, Dubiel SM, Sepioł B (2000) Mössbauer effect study of the phase separation in the Fe-Cr system. *J Phys-Condens Mater* 12:6709–6717
3. Davis JR (1996) Stainless steels. ASM Specialty handbook. ASM International, Chagrin Falls, OH, USA
4. Jura J, Baudin Th, Mathon MH, Świątnicki W, Penelle R (2002) Microstructure and texture analysis in the cold-rolled austenitic-ferritic steel with duplex structure. *Mater Sci Forum* 408/412:1359–1364
5. Reick W (1993) Kaltumformung und Rekristallisation eines rostbeständigen ferritisch-austenitischen Duplex-Stahles. Ph.D. Thesis, Ruhr-Universität, Bochum, Germany
6. Skrzypek S, Kolawa E, Sawicki JA, Tyliszczak T (1984) A study of the retained austenite phase transformation in low alloy steel using conversion electron Mössbauer spectroscopy and X-ray diffraction. *Mater Sci Eng* 66:145–149
7. Wright SI, Adams BL, Kunze K (1993) Orientation imaging: the emergence of a new microscopy. *Metall Trans A* 24:819–831