Title: The Mossbauer spectroscopy studies of retained austenite

Author: J. Krawczyk, P. Bała, Janusz Frąckowiak

Citation style: Krawczyk J., Bała P., Frąckowiak Janusz. (2007). The Mossbauer spectroscopy studies of retained austenite. "Archives of Materials Science and Engineering" (Vol. 28, iss. 10 (2007), s. 633-636).

Archives of Materials Science and Engineering

Volume 28
Issue 10
October 2007
Pages 633-636

International Scientific Journal
published monthly as the organ of
the Committee of Materials Science
of the Polish Academy of Sciences

The Mössbauer spectroscopy studies of retained austenite

J. Krawczyk*[,a], P. Bała [a], J. Frąckowiak [b]
[a] Faculty of Metals Engineering and Industrial Computer Science, AGH-University of Science and Technology, ul. Mickiewicza 30, 30-059 Kraków, Poland
[b] Faculty of Computer and Materials Science, University of Silesia, ul. Uniwersytecka 4, 40-007 Katowice, Poland
* Corresponding author: E-mail address: jkrawczy@metal.agh.edu.pl

Received 18.06.2007; published in revised form 01.10.2007

ABSTRACT

Purpose: of this paper: This paper completes the knowledge concerning the mechanisms of destabilization and properties of retained austenite. Investigations were performed on 120MnCrMoV8-6-4-2 steel, which was designed in 1998, in Phase Transformations Research Group of Department of Physical and Powder Metallurgy at the Faculty of Metals Engineering and Industrial Computer Science at AGH University of Science and Technology in Krakow.

Design/methodology/approach: The samples of investigated steel were austenitized at the temperature of 900ºC and hardened in oil. Next, three from four samples were tempered. Tempering consisted of heating the samples up to chosen temperatures with a heating rate of 0.05ºC/s and, after reaching desired temperature, fast cooling. CEMS technique was applied for Mössbauer studies.

Findings: Stabilized by heating up to 80ºC retained austenite, in the result of mechanical destabilization, transforms into low-temperature tempered martensite, with the structure of low bainite (into the structural constituent in which ε carbide exists).

Research limitations/implications: The influence of the temperature, up to which the samples were heated during tempering, on the mechanical stability of retained austenite and on the products of its transformation, was determined.

Practical implications: Changes occuring in retained austenite during tempering of steel of high hardenability (hardness), developed for potential applications on tools of enhanced wear resistance, were described.

Originality/value: Mössbauer spectroscopy was applied not only for qantitative analysis of retained austenite, but also to analyze the values of quadrupole splitting and isomeric shift, what resulted in significant conclusions concerning the changes in its chemical composition, microstructure, and the level of stresses being present in it.

Keywords: Mössbauer spectroscopy CEMS; Steel; Retained austenite; Carbides

METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

1. Introduction

Mössbauer spectroscopy has already found a wide range of applications in the research concerning the physical metallurgy of iron-based alloys. An example of such research is an application of his technique in the analysis transformation of retained austenite in low-alloy steel [1]. However, in work [2], using Mössbauer spectroscopy, thermal stability of metastable austenite, present in fast-crystallized tool steel obtained by powder metallurgy method, was described. The analysis of Mössbauer spectrum allowed to determine the austenite transformation temperature range (618 ÷ 643°C). Similar, in work [3] an attempt to describe, using Mössbauer spectroscopy, an influence of deformation and the temperature of sub-zero treatment on the stability of retained austenite in low-carbon Cr-Ni-Mo steel.

Mentioned above works as well as many other investigations [4-12] showed, that Mössbauer spectroscopy gives a possibility to achieve new informations, which may help more detailed interpretation of the phenomena connected with transformations ongoing during tempering of hardened steel. Even though dilatometric, microscopic and mechanical investigations enabled discussing these issues [13-17], because of some restrictions of these methods they were not able to explain all of the mechanisms connected with phase transformations, giving only suggestions for choising one of their possible interpretations.

This work presents the results of investigations using Mössbauer spectroscopy technique and their interpretation concerning retained austenite and its transformation during tempering in relation to previously conducted dilatometric, microscopic and mechanical investigations [13].

2. Test material

The research was conducted on a new high-carbon alloy steel with the chemical composition given in Table 1.

Table 1.
Chemical composition of the investigated steel

mass %								
C	Mn	Si	P	S	Cr	Mo	V	Al
1.22	1.93	0.19	0.018	0.02	1.52	0.36	0.17	0.04

3. Experimental procedure

Samples, taken from investigated steel, were austenitized at the temperature of 900ºC and hardened in oil. Austenitizing time was 20 minutes. Next, three from four samples were tempered. Tempering consisted of heating the samples up to chosen temperatures with a heating rate of 0.05ºC/s and fast cooling after reaching desired temperature.

Sample No. 1 was left in as-hardened state. After hardening, sample No. 2 was heated to 80ºC, sample No. 3 was heated to 210ºC and sample No. 4 was heated to 350ºC. The temperatures up to which investigated samples were heated were selected in a way enabling precipitation of ε carbide in sample No. 2 during tempering. Sample No. 3 was heated up to temperature at which precipitation of ε carbide was finished and cementite started to precipitate without starting a transformation of retained austenite. Temperature, up to which sample No. 4 was heated after hardening was selected as a temperature corresponding to the finish of transformation of retained austenite.

All mentioned above temperatures were selected basing on CHT diagram (Fig. 1) published in work [17].

In this study Conversion Electron Mössbauer Spectroscopy (CEMS) with gas detector, filled with 98% He + 2% Ar, under pressure of 0.9 at., was applied. A Mössbauer source was ^{57}CoRh of activity of 10 mCi. Application of CEMS technique allowed investigating surface layers of thickness of about 100 nm. Two sides of each sample were investigated, one of which was ground and second one was polished after grinding.

4. Research results and discussion

Basing on the results of the investigations using TEM [13] it could be noticed, that directly after hardening from 900ºC the microstructure of studied steel consists of martensite (partly twinned), retained austenite and spheroidal particles of alloyed hypereutectoid cementite, undissolved during austenitizing.

The analysis of the intensity of individual component spectrums may be used for determination the amount of phase constituents, and for analysis of qualitative changes of the quantity of particular phase constituents during steel tempering in particular.

Basing on the analysis of hyperfine magnetic field and on previosly pefrormed investigations [13,17], conducted with different techniques, it is possible to determine from which phases particular spectrum comes from. The analysis of hyperfine magnetic field (Zeeman sextets) allowed distinguishing component spectrums coming from ^{57}Fe atoms, existing in the structure of martensite (or ferrite in the case of higher tempering temperatures), in the structure of ε carbide, and in cementite, independently precipiteted during tempering. A component spectrum was also identified, characterized by single peak (without quadrupole or Zeeman splitting) as coming from precipitations of alloyed hypereutectoid cementite (which is paramagnetic), undissolved during austenitizing.

This analysis allowed to distinguish component spectrum of quadrupole splitting, coming from ^{57}Fe atoms, and located in the structure of retained austenite.

A volume fraction of retained austenite (and the source intensity applied) did not allowed for splitting the spectrum coming from this austenite into components coming from ^{57}Fe atoms, having C atoms in their closest and these not having. It may influence the differences in the intensity of this component spectrum, what may in some extent influence estimation of the volume fraction of this phase. It makes difficult to analyse the carbon content existing in retained austenite. However, it can be stated, that in as-hardened sample retained austenite is characterized by much lower resistance to the mechanical destabilization than in the sample tempered at 80ºC (Fig. 2). A decrease of the intensity of spectrum coming from ^{57}Fe atoms existing in retained austenite, measured on ground surface of sample tempered at 80ºC in relation to as-hardened sample is most probably connected with a fact, that deformation occuring during grinding may destabilize even chamically stabilized during heating up to 80ºC retained austenite. Smaller amount of retained austenite on the surface of polished as-hardened sample, as compared to the ground surface of this sample, should be associated with high nonuniformity of stresses introduced during grinding into the surface of this sample. It is connected with the fact, that only from certain level of stresses, retained austenite still existing after hardening, undergoes mechanical destabilization. Polishing introduced smaller stresses into the analyzed by Mössbauer spectroscopy surface of sample, but in larger volume exceeds the value causing destabilization of retained austenite.

Smaller amount of retained austenite in the sample tempered at 210ºC (both on polished surface and ground surface), as compared to the sample tempered at 80ºC, can be associated with initiation of $\gamma - \alpha$ transformation, or with the difference in stability of retained austenite after tempering at 80 and 210ºC. It can also be noticed, that changes of retained austenite are very small and most probably they are within the confines of the measuring error. After tempering at 350ºC the component Mössbauer spectrums coming from retained austenite could not be noticed.

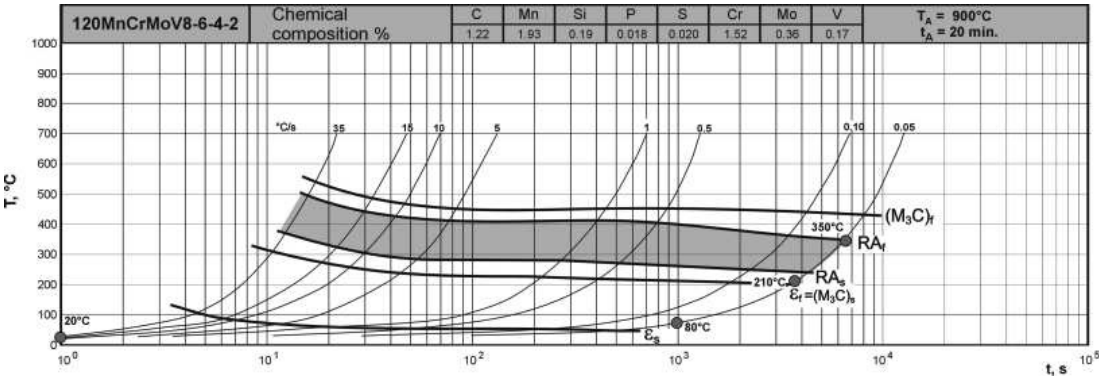

Fig. 1. Continuous heating transformations (CHT) diagram for investigated steel. After heating to the red point (marked on the 0.05°C/s curve) the CEMS was made [17]

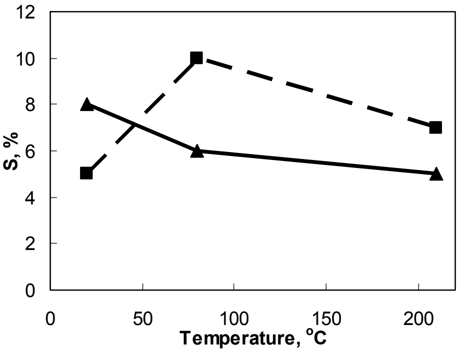

Fig. 2. Changes of intensity (S) of component Mössbauer spectrum coming from retained austenite: ■ - polished surface, – ground surface

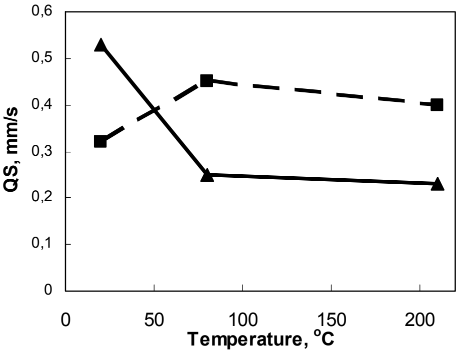

Fig. 3. Changes of quadrupole splitting (QS) of component Mössbauer spectrum coming from retained austenite: ■ - polished surface, ▲ – ground surface

The measurements performed with application of Mössbauer spectroscopy on the surface of ground as-hardened and tempered at 80ºC sample showed the presence of spectrum coming from ^{57}Fe atoms, located in the structure of ε carbide. It confirms previous observations, showing that in this temperature range the transformation of a part of retained austenite into low-temperature tempered martensite, with ε carbide in the structure, proceeds and that this transformation is strain induced (stresses). Better preparation of the surface of the sample (polishing) does not initiate such transformation.

No spectrum coming from ε carbide was noticed on the gruond surface or on the ground and polished surface of as-hardened sample. This confirms, that a part of retained austenite existing on the surface of the sample transforms into martensite. Therefore, it can be noticed, that heating as-hardened sample up to the temperature as low as 80ºC causes changes in the chemical composition of retained austenite.

Changes of values of quadrupole splitting (QS) – that is electrical fields gradients in the locations of ^{57}Fe atomic nucleuses – with the temperature, at which the samples of investigated steel were tempered, for retained austenite is shown on Figure 3.

The analysis of component spectrums coming from retained austenite proves, that in the result of preparing the surface of investigated samples (stresses and strains introduced), these areas of retained austenite transform where ^{57}Fe atoms are in a state of smallest symetry of crystal structure. Considering the above statement, it can also be noticed, that if more retained austenite transformed in the result of mechanical destabilization (the larger stresses acted), then retained austenite, remained in the surface layer, has more symmetrical lattice, what means that ^{57}Fe atoms existing in its structure have smaller value of quadrupole splitting.

Compering the values of quadrupole splitting on ^{57}Fe atomic nucleuses, measured on the sample tempered at 80ºC and sample tempered at 210ºC, it can be noticed, that decreasing degree of supersaturation of martensite with carbon decreases the level of stresses in retained austenite, because the value of quadrupole splitting decreases in this range with an increase of tempering temperature. The changes mentioned above may be observed both in the case of measurements performed on ground surfaces of the samples and also on these polished after grinding surfaces.

Figure 4 shows changes of the values of isomeric shift (IS) for ^{57}Fe atomic nucleuses, located in retained austenite. As it can be seen, more careful (more delicate) preparation of the sample surface, especially in the case of stabilized during tempering austenite, causes smaller isomeric shift for ^{57}Fe atomic nucleuses. It can be assumed, that such austenite will be characterized by smaller stresses.

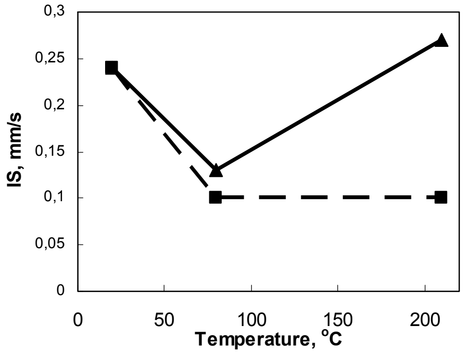

Fig. 4. Changes of isomeric shift (IS) of component Mössbauer spectrum coming from retained austenite: ■ - polished surface, – ground surface

5. Conclusions

Obtained results lead to the following conclusions:

retained austenite after hardening is less resistant to mechanical destabilization than after tempering at 80ºC,

stabilized by heating up to 80°C retained austenite undergoes, in the result of mechanical destabilization, transformation into low-temperature tempered martensite of the structure of low bainite, in which ε carbide exists,

retained austenite in hardened sample transforms into martensite in the result of mechanical destabilization,

heating as-hardened sample up to the temperature of 80ºC causes changes in the chemical composition of retained austenite,

if more retained austenite transformed in the result of mechanical destabilization (the larger stresses acted), then retained austenite, remained in the surface layer, has more symmetrical lattice.

References

[1] S. Skrzypek, E. Kolawa, J. A. Sawicki, T. Tyliszczak, A study of the retained austenite phase transformation in low alloy steel using conversion electron Mössbauer spectroskopy and X-ray diffraction, Materials Science and Engineering 66 (1984) 145-149.

[2] P. Grgac, R. Moravick, M. Kusy, I. Toth, M. Miglierini, E. Illekova, Thermal stability of metastable austenite in rapidly solidified chromium-molybdenum-vanadium tool steel powder, Materials Science and Engineering A 375-377 (2004) 581-584.

[3] P.D. Bilmes, M. Solari, C.L. Liorente, Characteristics and effects of austenite resulting from tempering of 13CrNiMo martensitic steel weld metals, Materials Caracterization 46 (2001) 285-296.

[4] Soon-Ju Kown, Sei J. Oh, Joo Hag Kim, Sangho Kim, Sunghak Lee, Mössbauer analysis of heat affected zones of an sa 508 steel weld, Scripta Materialia 2, (1999) 131-137.

[5] V.G. Gavriljuk, Decomposition of cementite in pearlitic steel due to plastic deformation, Materials Science and Engineering A345 (2003) 81-89.

[6] R. Ilola, V. Nadutov, M. Valo, H. Hänninen, On irradiation embrittlement and recovery annealing mechanisms of Cr-Mo-V type pressure vessel steels, Journal of Nuclear Materials 302 (2002) 185-192.

[7] V.A. Shabashov, L.G. Korshunov, A.G. Mukoseev, V.V. Sagaradze, A.V. Makarov, V.P. Pilyugin, S.I. Novikov N. F. Vildanova. Deformation-induced phase transitions in a high-carbon steel. Materials Science and Engineering A346 (2003) 196-207.

[8] V. A. Shabashov, A. G. Mukoseev, V. V. Sagaradze, Foration of solid solution of carbon in BCC iron by cold deformation, Material Science and Engineering A307 (2001) 91-97.

[9] A. G. Balanyuk, V. G. Gavriljuk, V. N. Shivanyuk, A. I. Tyshchenko, J. C. Rawers, Mössbauer study and thermodynamic modeling of Ee-C-N alloy, Acta Mater. 48 (2000) 3813-3821.

[10] E. J. Miola, S. D. De Souza, M. Olzon-Dionysio, D. Spinelli, C. A. Dos Santos, Nitriding of H-12 tool steel by direct-current and pulsed plasmas, Surface and Coatings Technology 116-119 (1999) 347-351.

[11] P. Budziński, P. Tarkowski, E. Jartych, A. P. Kobzev, Evolution of mechanical properties in tool steel implanted witch high energy nitrogen ions 63 (2001) 737-742.

[12] Y. Jirásková, J. Svoboda, O. Schneeweiss, W. Daves, F. D. Fischer, Microscopic investigation of surface layers on rails, Applied Surface Science 239 (2005) 132-141.

[13] P. Bała, The kinetics of phase transformations during tempering and its influence on the mechanical properties, PhD thesis, AGH University of Science and Technology, Cracow 2007. Promotor J. Pacyna (in Polish).

[14] P. Bała, J. Pacyna, J. Krawczyk, The kinetics of phase transformations during tempering in the new hot working steel, Journal of Achievements in Materials and Manufacturing Engineering 22 (2007) 15-18.

[15] P. Bała, J. Pacyna, J. Krawczyk, The kinetics of phase transformations during the tempering of HS18-0-1 high-speed steel. Journal of Achievements in Materials and Manufacturing Engineering 19 (2006) 19-25.

[16] P. Bała, J. Pacyna, J. Krawczyk, The kinetics of phase transformations during the tempering of HS6-5-2 high-speed steel. Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 47-50.

[17] J. Pacyna, P. Bała, T. Skrzypek, The kinetic of phase transformation during continuous heating from quenched state of new high-carbon alloy steel, Proceedings of the 13th International Scientific Conference „Achievements in Mechanical and Materials Engineering" AMME'2005, Gliwice-Zakopane 2005, 509-512.