Methodological approaches to the study of reverse martensitic transformation in metastable austenitic steels

To cite this article: D O Panov *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1014** 012042

Methodological approaches to the study of reverse martensitic transformation in metastable austenitic steels

D O Panov[1*], A I Smirnov[2], Y N Simonov[3], N D Stepanov[1], G A Salishchev[1]

[1] Laboratory of Bulk Nanostructured Materials, Belgorod State University, 85 Pobeda Str., 308015, Belgorod, Russian Federation
[2] Department of Materials Science in Engineering, Novosibirsk State Technical University, 20 Prospekt K. Marksa, 630073, Novosibirsk, Russia
[3] Department of Metal Science, Thermal and Laser Processing of Metals, Perm National Research Polytechnic University, 29 Komsomolsky prospekt, 614990, Perm, Russia

*Corresponding author: panov_d@bsu.edu.ru

Abstract. Methodological aspects of the high-resolution dilatometry analysis of the reverse α→γ-transformation in cold-worked metastable austenitic steel Fe-0.07C-18.7Cr-9.2Ni-0.6Ti-1.1Mn-0.4Si (wt.%) were considered. It was shown that the reverse α→γ-transformation occurs in the temperature range from A_S=520 °C to A_F=920 °C. Two consecutive stages corresponding to shear and diffusion transformations were revealed. An abnormal increase in the sample length of the program steel during the reverse α→γ-transformation was found. The recrystallization process also developed during austenitization and was accompanied by a decrease in length of the specimen.

1. Introduction

Dilatometry has established itself as one of the main methods for examination of phase transformations in steels and iron alloys [1–4]. The dilatometry method is based on measuring changes in length of a material under heating/cooling. The measurements provide information about phase transformations and structure changes, i.e. is able to capture all processes that have a volumetric effect. Advances in research equipment make it possible to identify extremely fast phase transformations or record very weak effects with high accuracy [4].

Mathematical analysis of dilatometric data, i.e. using the first derivative, increases the accuracy of determining the phase transformation temperatures critically [5,6]. However, high-resolution dilatometry has revealed that even the derivative often shows a superposition of several peaks from various transformations [6–8]. After proper analysis, the austenitization stages in dual-phase ferrite-perlite steels [9], the bainite-ferrite steels [7], and the martensitic steels [10] were revealed. In these materials, different transformations can occur in the same temperature range but in different micro-volumes. However, this approach has never been used in the case of the reverse α→γ-transformation during heating of cold-deformed metastable austenitic steels. Thus, the purpose of this work is to analyze the reverse α→γ-transformation in cold-worked metastable austenitic steel using high-resolution dilatometry.

2. Experimental

Metastable austenitic steel Fe-0.07C-18.7Cr-9.2Ni-0.6Ti-1.1Mn-0.4Si (wt.%) was used as the program material. Pure nickel was selected as the reference material. Before cold deformation, a rod of the program steel was heated to a temperature of 1050 °C, held for 2 hours and water quenched. Then the rod was deformed at room temperature using a radial forging machine to a true strain of 2.14.

Transmission electron microscopy was performed using a FEI Tecnai 20 G2 TWIN microscope at an accelerating voltage of 200 kV. Samples with a thickness of 300 microns were cut by an electric-discharge machine from the center of the rod in longitudinal and transversal sections. Then they were ground on both sides with abrasive paper with a decreasing grain size. Subsequent electrolytic thinning of disks with a diameter of 3 mm and a thickness of 100 microns was performed using TenuPol-5 in a mixture of 95% acetic acid (CH_3COOH) and 5% perchloric acid ($HClO_4$) cooled to -40 °C. The content of the α-phase was measured using a multifunctional eddy-current tester MVP-2M.

Dilatometry was performed using a Linseis R.I.T.A. L78 quenching dilatometer in a helium (purity of 99.9999%) atmosphere. Cylindrical samples with a diameter of 3 mm and a height of 10 mm were cut from the center of the rod. The long axis of the samples was aligned with deformation direction. The samples were heated to 1000 °C at a rate of 10 °C/s. Analysis of dilatometric curves was performed by obtaining the first derivative of dilatograms ($d(\Delta L)/dT$)) with subsequent peaks fitting in the Fityk software [11]. Asymmetric Gaussian curves were used for the approximation of the experimental results. The corresponding R-square values were ≥0.95.

3. Results and discussion

In the initial condition, the program steel had a lamellar dual-phase martensitic-austenitic structure, containing 62.5±0.9 % of deformation-induced martensite. In the longitudinal section, the grains were stretched along the axis of the rod (Figure 1a). Meanwhile, the grains had a fairly equiaxed shape (Figure 1b) with an average diameter of 240±10 nm in the transversal direction. Thus, it seems that the grains had a columnar shape. A more detailed description of the initial structure can be found elsewhere [12,13].

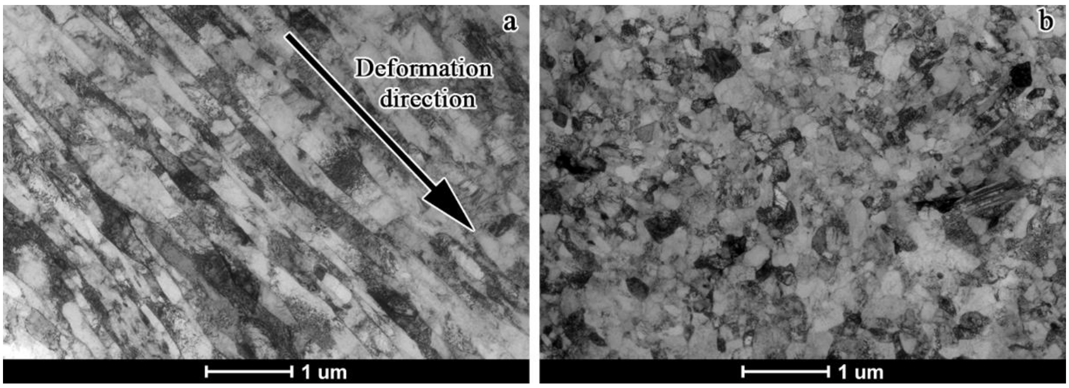

Figure 1. TEM bright-field images of a structure of the program steel in *a* longitudinal and *b* transversal section.

The dilatometric curve (ΔL) for the nickel sample was linear without any visible inflections. The first derivative of the dilatometric curve ($d(\Delta L)/dT$-curve) also did not demonstrate any evidences of the phase transformation (Figure 2a). At the same time, the ΔL curve obtained during heating of the program steel exhibits a series of inflections, associated with the development of the reverse α→γ-transformation (Figure 2b). The temperatures of the start (A_S) and finish of the transformation (A_F) were determined from the $d(\Delta L)/dT$. The respective values were A_S = 520 °C and A_F = 920 °C.

The $d(\Delta L)/dT$-curve in a A_F - A_S range is obviously the product of a superposition of several peaks from different events (Figure 2c). Fitting the peaks to asymmetric Gaussian curves helped to consider the effect of varying conditions during heating [10]. Note that the beginning and end of the peak on the *d(ΔL)/dT*-curve corresponds to the temperatures of the start and finish of the transformation, respectively; the peak area indicates the volume effect of the transformation [14]; and the maximum position corresponds to the highest speed of the process.

The performed analysis has allowed to identify three peaks in the temperature range of the α→γ-transformation (Figure 2c). Peaks #1 and #2 were associated with an increase in the length of the specimen, since they had upward orientation from the baseline. These peaks are likely direct products of the α→γ-transformation. A similar anomalous increase in the sample length during the reverse α→γ transformation was observed in AISI 304 steel [15]. In that case the volumetric expansion was associated with the effect of the gamma fiber texture ({111}<110> and {111}<112>) in the deformation-induce martensite. Furthermore, the α→γ-transformation by shear mechanism occurs at a lower temperature, while the activation of the diffusion mechanism usually requires higher temperatures [16]. Therefore, peak #1 corresponds to the shear transformation, and peak #2 – to the diffusion transformation. Peak #3 was oriented downward from the baseline, which corresponds to a decrease in the sample length. A similar effect was earlier associated with the development of recrystallization during heating of cold-deformed steel [17].

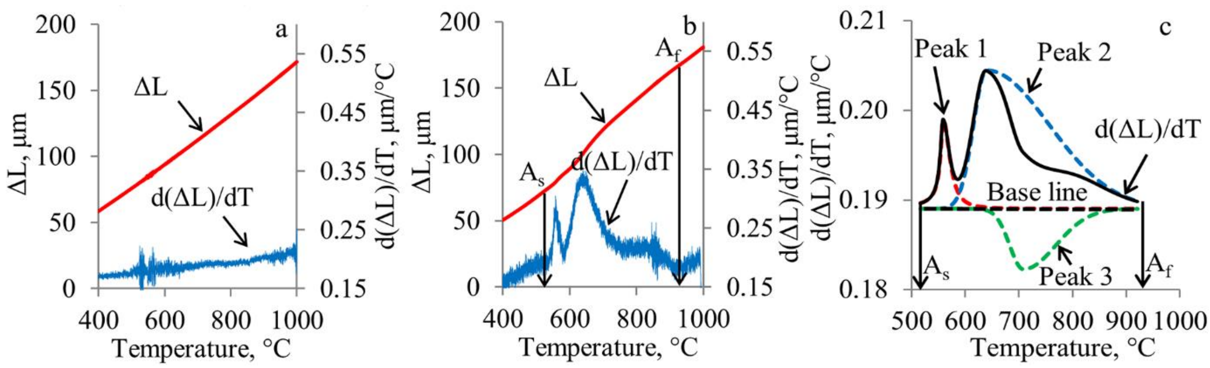

Figure 2. *a* dilatometric curve (*ΔL*) and *d(ΔL)/dT*-curve of the pure nickel sample and b the program steel; *c* the sketch explaining *d(ΔL)/dT*-curve for the program steel.

4. Conclusions

A method for analyzing the reverse α→γ transformation in cold-worked metastable austenitic steel Fe-Fe-0.07C-18.7Cr-9.2Ni-0.6Ti-1.1Mn-0.4Si (wt.%) using dilatometric curves was described. The reverse α→γ-transformation started at A_S=520 °C and finished at A_F=920 °C. Peaks from the reverse α→γ-transformation by the shear and diffusion mechanisms, as well as from the recrystallization process, were identified. The established approach allows us to develop the heat treatment regimes for cold-worked metastable austenitic steels to obtain the required structures and properties.

Acknowledgements

The authors gratefully acknowledge the financial support from the Russian Science Foundation Grant no. 20-79-10094.

References

[1] Bojack A, Zhao L, Morris P F and Sietsma J 2012 In-situ determination of austenite and martensite formation in 13Cr6Ni2Mo supermartensitic stainless steel *Mater. Charact.* **71** 77–86

[2] Wei R, Enomoto M, Hadian R, Zurob H S and Purdy G R 2013 Growth of austenite from as-quenched martensite during intercritical annealing in an Fe-0.1C-3Mn-1.5Si alloy *Acta Mater.* **61** 697–707

[3] Suh D W, Oh C S, Han H N and Kim S J 2007 Dilatometric analysis of austenite decomposition considering the effect of non-isotropic volume change *Acta Mater.* **55** 2659–69

[4] García De Andrés C, Caballero F G, Capdevila C and Álvarez L F 2002 Application of dilatometric analysis to the study of solid-solid phase transformations in steels *Mater. Charact.* **48** 101–11
[5] Li P, Li J, Meng Q, Hu W and Xu D 2013 Effect of heating rate on ferrite recrystallization and austenite formation of cold-roll dual phase steel *J. Alloys Compd.* **578** 320–7
[6] Oliveira F L G, Andrade M S and Cota A B 2007 Kinetics of austenite formation during continuous heating in a low carbon steel *Mater. Charact.* **58** 256–61
[7] Li D, Min Y, Wu X 2010 Calculation of austenite formation kinetics of copper-bearing steel during continuous heating *J. Iron Steel Res. Int.* **1** 62-6.
[8] Son Y Il, Lee Y K and Park K T 2006 Reverse transformation of ferrite and pearlite to austenite in an ultrafine-grained low-carbon steel fabricated by severe plastic deformation *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.* **37** 3161–4
[9] Pawłowski B 2012 Determination of critical points of hypoeutectoid steels *Arch. Metall. Mater.* **57** 957–62
[10] Panov D O and Smirnov A I 2017 Features of austenite formation in low-carbon steel upon heating in the intercritical temperature range *Phys. Met. Metallogr.* **118** 1081–90
[11] Wojdyr M 2010 Fityk: A general-purpose peak fitting program *J. Appl. Crystallogr.* 43 1126–8
[12] Panov D, Pertsev A, Smirnov A and Khotinov V 2019 Metastable Austenitic Steel Structure and Mechanical Properties Evolution in the Process of Cold Radial Forging *Materials* **12** 2058
[13] Panov D O, Smirnov A I and Pertsev A S 2019 Formation of Structure in Metastable Austenitic Steel during Cold Plastic Deformation by the Radial Forging Method *Phys. Met. Metallogr.* **120** 184–90
[14] Cherepin V T 1968 Eksperimental’naia tekhnika v fizicheskom metallovedenii (Kiev: Tekhnika) in Russian
[15] He Y, Zhang J, Wang Y, Wang Y and Wang T 2019 The expansion behavior caused by deformation-induced martensite to austenite transformation in heavily cold-rolled metastable austenitic stainless steel *Mater. Sci. Eng. A* **739** 343–7
[16] Aletdinov A, Mironov S, Korznikova G F, Konkova T, Zaripova R G, Myshlyaev M M and Semiatin S L 2019 Martensite-to-Austenite Reversion and Recrystallization in Cryogenically-Rolled Type 321 Metastable Austenitic Steel *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.* **50** 1346–57
[17] Liu G, Li J, Zhang S, Wang J and Meng Q 2016 Dilatometric study on the recrystallization and austenization behavior of cold-rolled steel with different heating rates *J. Alloys Compd.* **666** 309–16