

Micro-Compression Test of Thixoformed Austenite

MASEK Bohuslav^{1,a}, AISMAN David^{1,b}, JIRKOVA Hana^{1,c}
 and WURSTER Stefan^{2,d}

¹University of West Bohemia in Pilsen, Research Centre of Forming Technology – FORTECH,
 Univerzitní 22, CZ-30614 Pilsen, Czech Republic

²University of Leoben, Erich Schmid Institute of Materials Science, Jahnstrasse 12, 8700 Leoben,
 Austria

^amasekb@kmm.zcu.cz, ^bdaisman@vctt.zcu.cz, ^ch.jirkova@email.cz, ^dstefan.wurster@oeaw.ac.at

Keywords: Mini-thixoforming, semi-solid state, micro-compression test

Abstract: Thixoforming is an alternative forming method, by which intricate and complex-shaped products can be manufactured using a single production step. This technology allows a material's microstructure to be altered profoundly. Typical microstructure of steels processed in this manner consists of quasi-polyhedral austenite grains embedded in a ledeburite-carbide network. This type of microstructure was produced by processing the experimental material in this study: the X210Cr12 steel. Since austenite is a metastable component depending on oversaturation with a number of elements, its thermal and mechanical stability needs to be known. This information is required for further modification and enhancement efforts. In previous experiments, the thermal stability was tested by thermal exposure. In the present work, the behaviour of austenite was explored under mechanical load at room temperature in a micro-compression test. A single block of austenitic material was used for making a test specimen with the dimensions of $2.4 \times 2.2 \times 4.9 \mu\text{m}^3$. Its mechanical properties were measured and deformation stability was investigated using compressive deformation.

Introduction

In this study, the special process of mini-thixoforming [1] was used for semi-solid processing of material. In terms of its parameters, the process was derived from the conventional thixoforming method. It is used for forming very small amounts of metal. Thanks to this fact, the achievable heating and cooling rates are on the order of 100 K/s. The highly dynamic nature of the process and the transition through the semi-solid state promote the formation of unconventional microstructures with very interesting combination of both mechanical and physical properties. They are multi-phase structures, forming in part due to different concentrations of chemical elements in liquid and solid phases.

The X210Cr12 tool steel was used as the experimental material (Table 1). The material's initial microstructure consists of primary chromium carbides and globular cementite embedded in ferrite matrix (Fig. 1). A typical feature of this material is its low plasticity, which is why it is difficult to form by conventional methods. It is, however, suitable for semi-solid processing, as it has a wide freezing range.

Table 1. Chemical composition of experimental material

Element	C	Cr	Mn	Si	Ni	P	S
Content [wt%]	2.01	11.3	0.27	0.23	0.08	0.014	<0.001

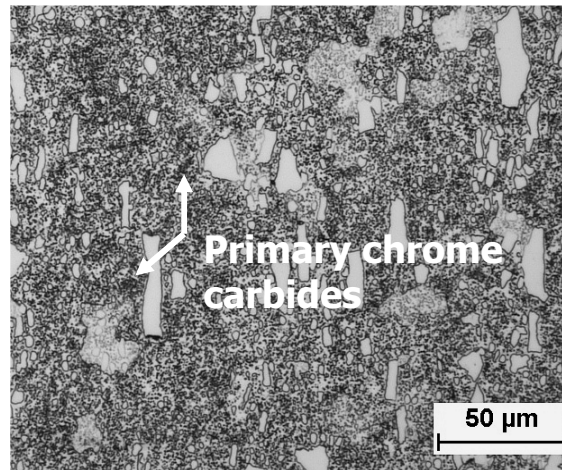


Fig. 1. Initial microstructure of experimental material

The material upon mini-thixoforming contains polyhedral austenite grains in carbide-austenite network. The total fraction of austenite in the microstructure, as measured by X-ray diffraction, exceeds 90%. The mean size of austenite grains, 12 – 14 μm , is considerably smaller than that of conventionally thixoformed materials. Since austenite in this type of steel is metastable at room temperature, its thermal stability had been examined in previous studies. In thermal exposure experiments, it proved to be stable up to about 500°C. Near that temperature, the austenite decomposed into troostite with very fine lamellae [2, 3]. Deformation stability of the material was examined and its mechanical properties were measured using micro-compression test. This test was developed for mechanical testing of a very small volume of material, such as thin films, metallic glasses, and others. In this case, the test was used for selective mechanical testing of properties of the austenite phase.

Experimental Details and Discussion of Results

The material for the microtest was obtained by mini-thixoforming at 1265 °C. The procedure included rapid solidification and cooling. Specimens for the microtest were taken from the processed material. In order to allow selective testing of mechanical properties of austenite without effects of the ledeburite network, a suitable region within a polyhedral austenite grain was identified using a scanning electron microscope. The test specimen was then cut out from this area using FIB. One side of the specimen remained attached to the processed block of material, which thus served as a tool and a handling fixture at the same time. After cutting, the specimen was polished by ion beam to achieve the best possible geometric accuracy (Fig. 2). The final dimensions of the test specimen were $2.4 \times 2.2 \times 4.9 \mu\text{m}^3$.

The compression test was carried out in an experimental device at the Erich Schmidt Institute in Leoben [4]. Force and displacement were recorded and the test was captured on video at the rate of 5 frames/s (Fig. 4). The tool velocity was 0.005 $\mu\text{m/s}$, which corresponded to a strain rate of $3.34 \cdot 10^{-3} \text{ s}^{-1}$. The compression continued up to the strain magnitude of $\phi = 0.77$. At this level, deformation became localised, continuing in visible slip bands.

The test was examined using the video recording and evaluated upon constructing the stress-strain plot. Slip bands, which formed at the last stage of loading, were clearly to be seen in the video sequence (Fig. 3). The bands spread across the cross-section of the specimen. The most intensive band formation, however, occurred at the base of the specimen, where the specimen was attached to the feedstock. This phenomenon was strengthened by the concentration of stress during loading and by the fact that spreading of the material was prevented.

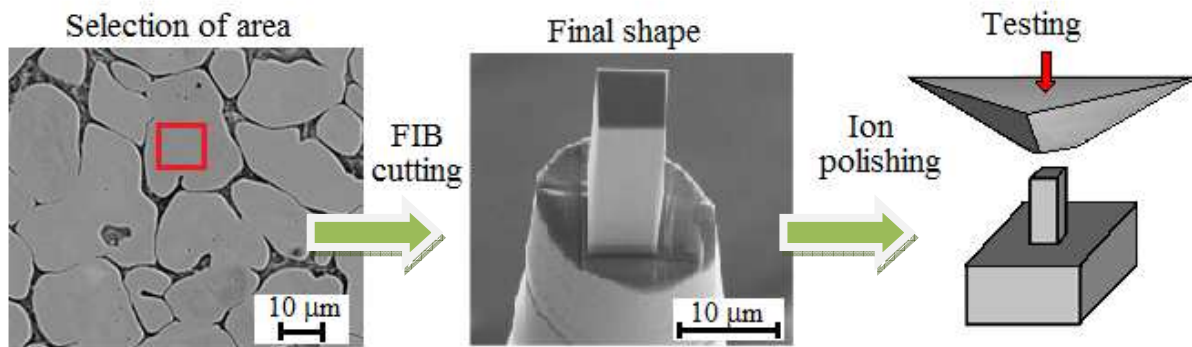
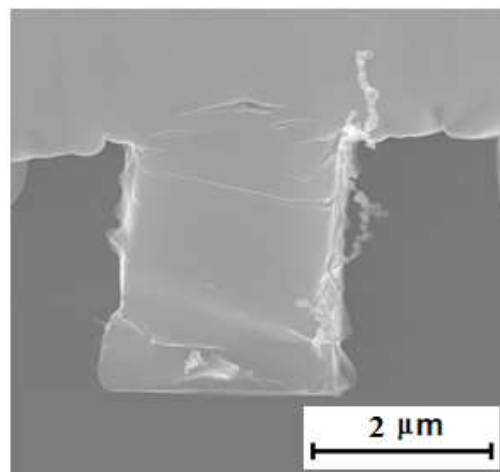
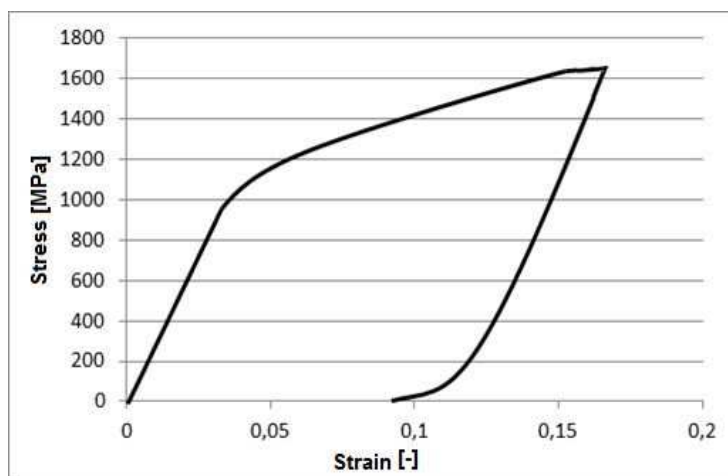


Fig. 2. Preparation for micro-compression test

Fig. 3. Test specimen upon the deformation of $\phi = 0.77$

The stress-strain plot shows a linear increase in stress up to the level of 1000 MPa. This represents a relatively high elastic resilience of the austenite phase. Above 1000 MPa, yielding occurs. Plastic deformation takes place in a uniform manner up to 1600 MPa. Above this level, slip bands began to form, causing the stress-strain curve to level off. The maximum force reached 8.84 μN at the reduction level of 1.49 μm , corresponding to the strength of 1667 MPa. At this reduction level, the test was suspended due to localisation of deformation. However, the test specimen did not fail at this point yet. Spreading was not evaluated, as the cross-section of the specimen was square-shaped.

Fig. 4. Stress-strain plot of the compression test of a cuboid-shaped specimen with dimensions of $2.4 \times 2.2 \times 4.9 \mu\text{m}$

Conclusion

Mini-thixoforming of X210Cr12 steel feedstock produced a microstructure consisting of ledeburite network with polyhedral grains of metastable austenite with a size of 12 – 14 μm . The stability of this type of microstructure and its behaviour during cold deformation were unknown. Knowledge of the behaviour of highly oversaturated austenite is required for further development and modification of these structures. For this reason, a compression test was conducted on a cuboid-shaped specimen with the size on the order of micrometres. The specimen was cut out from a single austenite block using FIB to allow testing of pure austenite without carbide network. In the course of the test, the specimen underwent extensive elastic deformation. Metastable austenite remained untransformed. With growing plastic strain, slip bands began to form. The localisation of deformation in slip band took place in stages. The compressive yield strength of the austenite phase exceeded 1000 MPa. Loading continued up to the stress level of 1670 MPa. At this point, deformation became considerably localised. However, the specimen did not fail even at this level, which could have happened due to local fractures caused by the plastic deformation ability being exceeded. Upcoming investigation efforts will be devoted to detailed examination of the state of microstructure at various stages of the loading process. The results will be used for controlled preliminary mechanical processing of microstructure prior to thermomechanical treatment in order to obtain new variants of thixoformed microstructure.

Acknowledgement

This paper includes results obtained within the project P107/11/J083 The Influence of Input Microstructure on Final Properties of Material Processed by Mini-Thixoforming. The project is subsidised from specific resources of the state budget for research and development.

References

- [1] D. Aisman, H. Jirkova, M. Behulova, B. Masek, Influence of Cavity Shape and Entry Hole on Mini-Thixoforming, in: J. Kliber (Ed.), Proceedings of 20th International Conference on Metallurgy and Materials, Tanger, Ostrava, 2011, pp. 210-215.
- [2] D. I. Uhlenhaut, J. Kradolfer, W. Puttgen, J. F. Löffler, P. J. Uggowitzer, Structure and properties of a hypoeutectic chromium steel processed in the semi-solid state, *Acta Materialia* 54 (2006) 2727–2734.
- [3] H. Jirkova, D. Aisman, B. Masek, Unconventional Structure of X210Cr12 Steel Obtained by Thixoforming, *Journal of Alloys and Compounds*. 504S (2010) 500-503.
- [4] D. Kiener, C. Motzb, G. Dehmb, Micro-compression testing: A critical discussion of experimental constraints, *Materials Science and Engineering: A*. Volume 505, Issues 1–2, 15 (2009) 79–87.