

Contents lists available at [ScienceDirect](http://www.sciencedirect.com/science/journal/09258388)

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

Metastable structure of austenite base obtained by rapid solidification in a semi-solid state

D. Aisman∗, H. Jirkova, L. Kucerova, B. Masek

University of West Bohemia in Pilsen, Research Centre of Forming Technology, Univerzitní 22, CZ – 306 14 Pilsen, Czech Republic

article info

ABSTRACT

Article history: Received 23 July 2010 Received in revised form 21 December 2010 Accepted 30 December 2010 Available online 4 January 2011

Keywords: Semi-solid processing Rapid cooling Metastable austenite

Material processing in a semi-solid state with rapid solidification is an innovative technology, which enables us to produce complex-shaped semi products in one operation. Unconventional properties and microstructures can be obtained in this way. Material processing in a semi-solid state has been used for materials with lower melting temperatures, particularly for Al alloys.

This paper concentrates on the development of new technologies for production of miniature thinwalled steel components with complicated shapes. Ledeburitic steel with 1.8% of carbon and 11% of chromium was chosen for this experimental study. This material was used to produce very small thinwalled semi products. From the initial structure consisting of primary and secondary carbides distributed in a ferrite matrix was obtained a microstructure with over 90% of metastable austenite after cooling from the semi-solid state. The main aim of this experimental program was to describe the effect of two different methods of heating to the semi-solid state. The first method used unique heating equipment, combining high frequency and resistance heating. The second method consisted of conventional heating in a furnace. The influence of the cooling rate on the development of the microstructure was investigated. If was found that both heating and cooling rates influence grain size and the size and the morphology of carbide network placed between the globular austenite grains.

Structure analysis was performed with the help of light microscopy, laser scanning confocal microscopy and scanning electron microscopy. EDX analysis was applied to determine chemical profiles of phases and X-ray diffraction phase analysis was used to establish volume fraction of austenite in the final microstructure. Characterisation of individual structural components was further completed by hardness measurements.

© 2011 Elsevier B.V. All rights reserved.

1. Introduction

Forming in a semi-solid state, so called thixoforming, requires heating of the semi-product to a partially solid and partially liquid state where it is also formed. The volume fraction of liquid is usually between 40% and 60%. The formed semi product then has thixotropic behaviour, meaning that the system has high viscosity which drops rather quickly during shear loading. Once the system is unloaded, its viscosity tends to return to its original high value [\[1\].](#page-3-0) Materials processed by this technology usually possess very unconventional microstructures. It can be demonstrated on high-alloyed steel whose microstructure after semi-solid state processing consisted of globular polyhedral grains of metastable austenite surrounded by a lamellar carbide network.

2. Experimental material

Tool steel X210Cr12 ([Table 1\)](#page-1-0) was chosen for this experimental program, because the properties of this steel are suitable for semi-solid processing. This material has low formability and machinability by conventional methods, as it has quite a high chromium content which makes it very hard and brittle. The initial microstructure was made of a ferritic matrix with globular cementite and primary chrome carbides [\(Fig. 1\).](#page-1-0) The hardness after annealing was 207 HV10. A suitable forming temperature was determined to be 1290 ◦C [\(Fig. 2\).](#page-1-0) The 40% content of the liquidus phase at this temperature was calculated in JMatProg and verified by comparing with other authors [\[2,3\].](#page-3-0)

3. Results and discussion

3.1. Thixoforming with combined high frequency and resistance heating

3.1.1. Experimental set-up

New equipment for thixoforming which uses the principle of side extrusion of semi-solid material was designed on the basis of semi-solid forming [\(Fig. 3\)](#page-1-0). An integral part of the set-up is also a special high frequency resistance heating unit allow-

[∗] Corresponding author. Tel.: +42 0377 63 8330; fax: +42 0377 63 8052. E-mail address: daisman@vctt.zcu.cz (D. Aisman).

^{0925-8388/\$ –} see front matter © 2011 Elsevier B.V. All rights reserved. doi:[10.1016/j.jallcom.2010.12.211](dx.doi.org/10.1016/j.jallcom.2010.12.211)

Fig. 1. Initial microstructure of X210Cr12 steel.

Fig. 2. ThermoCalc equilibrium simulations of X210CrW12 steel as pseudo-binary cut [\[5\].](#page-3-0)

ing us to reach and control the required temperatures with high accuracy. This heating principle offers a much higher heating gradient than any other method. The highest heating rate is 200 \degree C per second. Heating of the material is done directly inside a form and thus complicated manipulation with semiliquid material is avoided and forming can be carried out at a precisely defined temperature. This form was produced from tita-

Table 1

Chemical composition of X210Cr12 as given by the producer.

Fig. 3. Diagram of the form, section.

nium alloy. The form was divided into an upper and a lower part to simplify manipulation with the tool itself and also with the tested material. The final shape of the product is determined by the shape of a cavity placed inside the body of the form.

3.2. Microstructures obtained by thixoforming

Heating to $1290 °C$ took about 1 min and then a 5 min hold at this temperature was subsequently carried out. Semi-solid material

Fig. 4. Structure of X210Cr12 steel after thixoforming, left: overview of the microstructure by light microscope, right: detail of lamellar network obtained by scanning electron microscope.

Fig. 5. Detail of austenite–ledeburite network after furnace heating and rapid cooling in water.

was then injected into a rectangular groove with the dimensions of 15 mm \times 5 mm \times 3 mm.

The microstructure after thixoforming consisted of polyhedral austenite grains lined with a network of ledeburite and austenite mixture ([Fig. 4\)](#page-1-0). The average size of the polyhedral austenite grains was 19µm and they formed about 72% of the microstructure. Repeated X-ray diffraction phase analysis measurements confirmed that the austenite volume fraction in the whole microstructure was about 96%. The volume fraction of the other phases reached 4%. This very high fraction of metastable austenite was caused not only by the chemical composition of the steel, but also by the high heating temperature and rapid solidification. The high quantity of austenite in the final microstructure is also in good agreement with predominantly austenitic microstructures obtained by semi-solid processing of similar hypoeutectic chromium steel [\[4\].](#page-3-0) The hardness of the material increased after processing to 332 HV10.

3.3. Conventional furnace heating of the steel to semi-solid state

Heating parameters are an important part of processing strategy and therefore conventional furnace heating to the semi-solid state was also tested. The influences of other heating method and the influence of cooling conditions on microstructure development were investigated in the second part of the experimental program. The semi product was furnace heated for 5 min to 1290 ◦C and then a 15 min hold at the temperature was carried out prior to either water or air cooling. Deformation was applied to the semi product just below the temperature of solidus to analyse the influence of deformation on microstructure refinement.

Fig. 7. Recrystallized structure after deformation.

Heating to the temperature of 1290 \degree C and subsequent rapid cooling in water produced a microstructure very similar to the ones achieved by thixoforming carried out in the form. The main difference was in austenite grain sizes. The average grain size after heating in the furnace reached $46\,\mu$ m which is in a good agreement with grain size measured by other authors in a similar material [\[5\]. H](#page-3-0)owever, grain size after forming in the closed form was only 19 \upmu m. This difference can be explained by different heating rates and also by refinement of the grains during intensive forming. The space between polyhedral grains was filled again by fine austenite–ledeburite mixture (Fig. 5). The hardness of globular austenite grains was 310 HV 0.05 and the carbide network reached a hardness of 708 HV 0.05.

The effect of free cooling in air was further investigated and the material was heated in the furnace to 1290 ◦C and then cooled freely in air.

The resulting microstructure consisted again of coarse austenitic grains of practically the same size as after quenching. The lamellar net was not as fine as in the previous case (Fig. 6). However, globular grains were in this case more markedly lined than after quick cooling and very fine nuclei of troostite were found in the grain areas. Globular grains reached a hardness of 308 HV 0.05, which corresponds to the results after water cooling. The hardness of the carbide network slightly decreased to 564 HV 0.05, which was 144 HV0.05 less in comparison with the water-cooled state.

The next processing used the same furnace heating to 1290° C with additional deformation just below the solidus curve temperature. This deformation was carried out as an upsetting of a cylindrical specimen with true logarithmic deformation $\varphi = 0.7$. Metallographic evaluation showed that intensive grain refinement and austenite grain recrystallization occurred in the area of severe

Fig. 6. Structure after furnace heating to semi-solid state with subsequent air cooling. Left: microstructure overview by light microscopy, right: detail of carbide network between austenite grains obtained by scanning electron microscopy.

plastic deformation due to absorbed deformation energy ([Fig. 7\).](#page-2-0) Average grain size of recrystallized grains reached 12 μ m, while non-recrystallized austenite grains were prolonged by the deformation. Applied deformation was however not big enough to break the carbide network, which surrounded the austenite grains prior to deformation. The network was only elongated along the newly shaped boundaries of the austenite grains.

4. Conclusions

The above experiment proposed new possibilities for semi-solid processing of steels. This processing resulted in the stabilisation of the austenitic microstructure at room temperature due to the rapid solidification process. The method can be used even for chromium alloyed steel with ledeburitic–carbidic original microstructure. The volume fraction of austenite in the final microstructure reached in this case 96%.

Conventional furnace heating of the same material required several times longer heating times which, together with the absence of deformation, supported undesirable coarsening of austenite grains (in comparison with thixoforming). The cooling rate on the other hand did not influence austenite grain size but rather the size and morphology of the lamellar austenite–ledeburite network. Rapid heat removal refined the network, while the decrease of the cooling rate made the network coarser and changed its morphology. Small troostite areas were found at the austenite grain boundaries after the slower cooling. Application of deformation at the temperature under the solidus curve significantly refined the microstructure and supported recrystallization of the austenite grains.

Acknowledgement

This paper includes results obtained within the project 1M06032 Research Centre of Forming Technology.

References

- [1] W. Püttgen, W. Bleck, G. Hirt, H. Shimahara, Thixoforming of steels a status report, Advanced Engineering Materials 9 (4) (2007) 231–245.
- [2] B. Hallsted, Use of calphad thermodynamics to simulate phase formation during semi-solid processing, in: G. Hirt (Ed.), The 10th International Conference on Semi-Solid Processing of Alloys and Components, Aachen, September, Trans Tech Publications Ltd., Zurich, 2008, ISBN 3-908451-59-0, pp. 641– 646.
- [3] D. Aisman, H. Jirkova, B. Masek, Forming technology of small parts in semi-solid state, in: Proceedings of the 20th International DAAAM Symposium Intelligent Manufacturing & Automation: Theory, Practice & Education, vol. 20, 2009, No. 1, ISSN 1726-9679, ISBN 978-3-901509-70-4, pp. 1895–1896.
- [4] W. Puttgen, B. Hallstedt, W. Bleck, P.J. Uggowitzer, On the microstructure formation in chromium steels rapidly cooled from the semi-solid state, Acta Materialia 55 (2007) 1033–1042.
- [5] D.I. Uhlenhaut, J. Kradolfer, W. Puttgen b, J.F. Loffler, P.J. Uggowitzer, Structure and properties of a hypoeutectic chromium steel processed in the semi-solid state, Acta Materialia 54 (2006) 2727–2734.