MINIATURIZATION IN THIXOFORMING

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Innovative technological processes can help us to obtain unconventional structures with specific mechanical properties, even from conventional materials. Forming metal alloys in the semi-solid state is one of many ways. With thixoforming the tool steel X210Cr12 was treated with rapid solidification from the liquid phase. The microstructure after processing in the semi-solid state is 96% globular austenite formations surrounded by a carbide network. The aim of the experiment was to test the new technological process and to describe the obtained microstructure and its stability. The stability of the microstructure was tested under various thermal constraints and mechanical loading. The hardness of the austenite component after thixoforming was about 320 HV 0.05; hardness of the carbide network was about 550 HV 0.05.

Keywords
thixoforming, Semi-Solid forming, rapid cooling, metastable austenite, tool steel

1. Introduction
Utilization of the thixo-properties of materials has spread into many areas of human activities and many different fields. The applications using the thixostate are commonly used in the building industry, in painting materials, foundry moulding materials, chemistry, the food industry, etc.

The first published experiments with the utilization of thixo-properties of metals were carried out in 1972. These experiments were done with tin-lead alloys at a temperature interval between solidus and liquidus, thus in a semi-solid state. These properties almost immediately became the centre of attention of many research groups, which investigated the hidden potential of this method. Thixoforming combines the advantages of casting and forming thus enabling the production of components with very complicated-shaped designs [Püttgen 2007], [Bührig_Polaczek 2007] (Fig. 1).

The fundamentals of the process is forming of the semi-product which is, after heating to processing temperature, partially in a solid and partially in a liquid state. Heating temperatures of the semi-product are usually higher than for common processing methods of the same material. This is the reason why the first experiments were aimed at materials with lower solidification temperatures. They were particularly aluminium or zinc alloys. Later it was possible to direct the research in the field of materials with higher solidification temperatures [Dziallach 2008]. Among these materials were different alloys of copper, nickel and cobalt. Still later, at the turn of eighties and nineties, the first steel semi-products were prepared by thixoforming. The first steels used were predominantly high alloyed steels, such as X105CrMo17, X5CrNi18–10 (Fig. 2). These steels were used mainly because their processing temperature for semi-solid forming is the lowest possible one for steels. The temperature interval between liquidus and solidus is relatively wide and their microstructure is suitable for plastic material flow with low resistance. Later experiments used also other kinds of steel, for example C60, C80, 100Cr6, etc. The research has intensively tried to obtain information about semi-solid state parameters and also about the optimal chemical composition of the material used [Püttgen 2007]. The aim was particularly to obtain a globular microstructure (Fig. 2).

Since the first research concerning the forming of steels in a semi-solid state, which were published at the beginning of 1980s, a lot of interesting results have been published. However, it is very important to master all the technological parameters of the process and control them with relatively small tolerances before the application of this process to routine industrial production is possible. The choice of materials and design of the tools is just as important as the technological parameters, as they have to endure extreme conditions, in particular, high temperature shock loading, high contact temperature and abrasion. These are the main technical reasons why this technology has not yet been employed on a large scale in mass-production. The forming process in a semi-solid state is, however, so interesting from the point of view of the shape variations of the product or resulting microstructures, that many innovative variations of this unconventional technology can be expected in the future.

Figure 1. An example of a product made by thixoforming from 49MnVS3 steel [Hirt 2009]

Figure 2. Application area of different technological processes in phase diagram of X210CrW12 steel with examples of typical structures
2. Mini-thixoforming
A new, as yet unpublished, method for manufacturing small components in the semi-solid state is ‘mini-thixoforming’ (Fig. 3, Fig. 4). This procedure was developed at the Research Centre of Forming Technology at the University of West Bohemia. Mini-thixoforming differs from conventional thixoforming in that it is necessary to develop new approaches for managing the temperature fields in the small volume of the material. This means a range of volumes up to one cubic centimetre. Minimum temperature deviations must be ensured throughout the heating process to obtain even distribution of temperature throughout the entire volume of the semi-product. Another advantage of mini-thixoforming is the very rapid solidification from the semi-liquid state after deformation. This means that unconventional structures can be obtained even with commonly used industrial materials, which have an interesting combination of not only mechanical but physical properties. These are multi-phase structures which are a result of, among other things, uneven distribution of chemical elements in the liquid and solid phases. The liquid phase is enriched by alloying elements during the thixoforming process which is together with high carbon content very important for following phase transformations. The solid phase usually consists of polyhedral austenitic grains with a lower content of the alloying element [Dziallach 2008].

For further development of the process it is important to precisely understand the principles, describe the origins of the structure and describe the individual structural components and their properties. It can be expected that the spectrum of materials used will be broadened to include non-conventional alloys with totally exotic structures.

3. Steel X210Cr12 minithixoforming
An experimental material, the X210Cr12 steel was chosen (Tab. 1). It is the steel of ledeburite type with a high content of chromium dedicated for tools used in cold forming. This steel is characterised by its difficult formability and machinability by conventional technologies. The microstructure consists of a ferritic matrix with globular cementite and primary carbide chrome in annealed state. The initial microstructure was formed of a ferritic matrix with globular cementite and primary chrome carbides (Fig. 3). The Martens hardness was determined to be 10182 N/mm² which corresponds to 1560 Vickers hardness. Small semi-products with thin walls were made using of thixoforming. The thin of walls were c. 1 mm (Fig. 4).

![Figure 3. Microstructure of the X210Cr12 steel in the initial state](image)

![Figure 4. An example of a demonstrator](image)

Table 1. Chemical composition of the X210Cr12 steel [wt. %]

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
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<tbody>
<tr>
<td>1.8</td>
<td>11</td>
<td>0.2</td>
<td>0.2</td>
<td>0.5</td>
<td>0.03</td>
<td>0.035</td>
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</tbody>
</table>

A suitable forming temperature was determined to be 1290°C. The 40% content of the liquidus phase at this temperature was calculated in JMatProg (Fig. 5).

![Figure 5. Fraction of liquid phase in relation to heating temperature](image)

3.1 Evaluation of microstructure
Metallographic analysis revealed that the structure after thixoforming consisted of polyhedral grains of austenite surrounded by fine carbide mesh. The microstructure could be obtained as a consequence of high pressure and rapid solidification during the forming process. By repeated X-ray diffraction phase analyses, the fraction of austenite in the structure was found to be 96%.

The fraction of ferrite and carbides reached 4%. The development of microstructure with such a high fraction of austenite resulted not only from the chemical composition but also from the high heating temperature, rapid solidification, rapid cooling and structure development under high applied pressure.

The structure of the obtained semi-product was homogenous. Only near the contact surface with the die, a thin layer formed by fine dendrites was developed due to the melt segregation and rapid solidification.

3.2 Stability of austenite
For evaluation of the stability of metastable austenite, experimental samples were subjected to thermal and mechanical loading (Tab. 2).

The initial assumption of austenite decomposition at the temperature of 200 °C was not confirmed. After a one hour hold at this temperature, no structural changes were identified by metallography. X-ray diffraction analysis affirmed the retention of the initial content of metastable austenite. A slight increase in hardness can be assigned to the precipitation of secondary carbides in the network surrounding the austenite.

The increase of tempering temperature to 350 °C with the holding time of one hour did not cause any visible changes of microstructure and the percentage of metastable austenite remained approximately the same (Fig. 6). Only the growth of hardness value to 461 HV10 was recorded which means a more than 20 % increase in comparison with the hardness after tempering at 200 °C. As well as in this case, the increase in hardness can be linked to the precipitation of secondary carbides.

The first visible changes of microstructure were observable at a temperature of 500 °C (Fig. 7). The growth of fine troostite occurred at grain boundaries. As the amount of troostite is small, the portion of metastable austenite remains almost 90 %. The hardness was not markedly changed in comparison with its value after exposure at the previous temperature.

The decomposition of metastable austenite in the structure was confirmed by the results of X-ray analysis according to which only 5% of austenite was determined. The austenite decomposition was
probably caused by the changes of carbon content. During the thermal exposure, carbon diffused to the grain boundaries and contributed to troostite development.

To test austenite stability at cryogenic temperatures, liquid nitrogen was used as a cooling medium. Metallurgical observations after this treatment revealed martensitic needles in the austenitic grains (Fig. 8). Despite the cryogenic conditions of the heat treatment, the temperature did not decrease significantly below the Ms temperature due to the high carbon content. Consequently, almost 90 % of metastable austenite remained in the structure. At cryogenic temperatures, the precipitation of secondary carbides in the network did not occur. This fact along with a high content of metastable austenite can explain relatively low final hardness. The stability of metastable austenite against plastic deformation was investigated at the room temperature. The deformation near the ultimate strength of material was applied. No changes in microstructure were noticeable using metallurgical observations. The decrease in the percentage of metastable austenite to 85 % was detected by X-ray diffraction analysis. It can be supposed that the drop in metastable austenite fraction was caused by deformation induced martensitic transformation. The phase transformation and deformation hardening represent probably the reasons for the hardness increase to 421 HV10. This value is 89 HV10 higher in comparison with material hardness in the initial state after thixotropic processing.

4. Conclusions
This experiment proved that very small parts with different final shapes can be produced from conventional X210Cr12 steel by the semi-solid forming process.

Thixoforming was performed in a titanium die cavity. The shapes of the demonstrator were varied. Very fine but otherwise typical thixoforming microstructures were obtained as a result of the combination of rapid solidification, deformation and a suitable chemical composition of steel. The microstructure was composed of polyhedral metastable austenitic grains surrounded by a ledeburite network. Austenite fraction was over 95 % and the hardness reached 332 HV10.

Thermal stability tests showed a high stability of austenite up to a temperature of 500 °C. During heating at temperatures above 500 °C and subsequent cooling to room temperature, metastable austenite decomposed and a special structure occurred which possessed high hardness of 757 HV10 in contrast to the initial state after thixoforming with a hardness of 332 HV10.

Martensite transformation temperature was reached during exposures in the range of cryogenic temperatures and therefore martensite needles appeared in the austenite.

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References

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<table>
<thead>
<tr>
<th>Material treatment</th>
<th>Percentage of metastable austenite [%]</th>
<th>Hardness HV 10</th>
</tr>
</thead>
<tbody>
<tr>
<td>annealed state</td>
<td>–</td>
<td>203</td>
</tr>
<tr>
<td>After thixoforming</td>
<td>96</td>
<td>332</td>
</tr>
<tr>
<td>350°C/1h</td>
<td>96</td>
<td>461</td>
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<td>757</td>
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<tr>
<td>-196°C/1h</td>
<td>89</td>
<td>366</td>
</tr>
<tr>
<td>Cold deformation</td>
<td>85</td>
<td>421</td>
</tr>
</tbody>
</table>

Tab. 2. Strategies of heat treatment with the final fraction of single phases

Figure 6. The detail of carbide-austenite mesh, 350°C/1 hour

Figure 7. Formation of fine troostite layer at the expense of globular formations at exposure temperature of 500°C

Figure 8. Martensitic needles after treatment for 1 hour in liquid nitrogen