

MINI-THIXOFORMING OF UNCONVENTIONAL STEELS PRODUCED BY POWDER METALLURGY

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Abstract

Mini-thixoforming is a method of processing metals at temperatures between the solidus and liquidus. Thanks to the small volume of feedstock, it allows very rapid heating and solidification rates to be achieved with great accuracy. With these features, it can even be used for processing materials with a very narrow freezing range which are otherwise unsuitable for conventional semi-solid processing techniques. Besides, the mini-thixoforming process can be used with difficult-to-form materials, such as high-alloyed powder steels. In these steels, unconventional microstructures can be produced by semi-solid processing. In the present experiment, powder steels with specific chemistries were used for obtaining such unconventional microstructures. The chemical compositions of these steels were selected with the purpose of exploring the effects of individual alloying elements, namely vanadium, chromium, molybdenum and tungsten. The materials in question were the CPM 15V, CPM S30V, K190 and CPM REX121 steels. Processing parameters were tentatively calculated for all of them. Semi-solid forming trials were then performed based on these calculations. Once the semi-solid processing parameters were verified experimentally, the required mould cavity filling and surface quality of demonstration products were achieved. All mini-thixoformed materials contained austenitic-martensitic matrix with various types of carbides.

Keywords: Mini-thixoforming, semi-solid state, powder steel, CPM 15 V, CPM S 30V

1. INTRODUCTION

Semi-solid processing is one of the unconventional processes which are based on forming between the solidus and liquidus [1]. Several routes known under the common name Semi-Solid Metal Processing (SSM) exist today [2]. One of them is the special mini-thixoforming process [3, 4]. In terms of its parameters, it is related to the conventional thixo-forming process. Unlike thixo-forming, however, it is used for processing very small volumes of metal on the order of tens of mm³ [5]. The process can thus be highly dynamic [6].

Today, commercial semi-solid metal processing is used for low-melting materials, primarily aluminium and magnesium alloys. Due to its technological complexity, thixo-forming of high-melting alloys is still under development. However, it offers great potential for processing difficult-to-form materials, including those which are difficult or impossible to process by conventional methods [7]. Such materials include, for instance, some high-alloyed powder steels. The present experiment focuses on working these steels in order to explore their potential for semi-solid forming and to find suitable forming parameters. Semi-solid processing invariably alters the microstructure of the material. In the course of the process, high temperature leads to partial melting and to intensive migration of chemical elements between individual phases [8]. Consequently, SSM processes can produce microstructures with morphologies and phase compositions different from those resulting from conventional forming methods. Professional literature still lacks an extensive description of the effects of individual elements on microstructural evolution during semi-solid processing of steels.

2. EXPERIMENTAL MATERIALS

Steels with high levels of carbon, chromium, vanadium, tungsten and cobalt were used in the experiment. The chemical compositions covered as wide range of concentrations as possible in order to map the effects of individual alloying elements (**Table 1**). These steels were the CPM 15V, CPM S30V, K190 and CPM REX 121 grades. All these steels were made by powder metallurgy.

Table 1 Chemical compositions of the steels used (wt. %)

	C	Cr	Mn	Mo	V	Si	W	Co
CPM 15V	3.4	5.25	0.5	1.3	14.5	0.9	-	-
CPM S30V	1.45	14	-	2	4	-	-	-
K190	2.3	12.5	0.4	1.1	4	0.1	-	-
CPM REX 121	3.42	3.99	0.51	5.21	9.18	-	9.75	8.71

The CPM 15V has the highest vanadium (14.5 %) and carbon contents (3.4 %) of the group. It offers long life under cold working conditions and good wear resistance thanks to a comparatively large amount of vanadium carbides. The CPM 15V steel is thus used for making dies, mandrels and parts of tools operating under extreme loads and in heavy-duty applications.

The second experimental steel was CPM S30V. Its carbon content is 1.4 %; its chromium level of 14 % is relatively high when compared to CPM 15V. It only contains 4 % vanadium. Thanks to its chemistry, it shows high corrosion and wear resistance. With its rounded vanadium carbides it can also provide adequate toughness. It is used for making knives and engineering parts.

The third experimental steel was K190 which shows good resistance to abrasive and adhesive wear. It has higher carbon content (2.3 %) than CPM S30V steel and almost identical levels of alloying elements. It was chosen for evaluating the effect of the carbon content on the microstructure evolution. CPM S30V is used predominantly for making pressing and precision cutting tools, deep drawing dies and metal cutting tools.

The last experimental steel is CPM REX 121, which is superior to the others in wear resistance, maximum hardness and hot hardness. It was chosen for its high levels of tungsten and cobalt. The effects of these elements on semi-solid processing have not been described in literature yet.

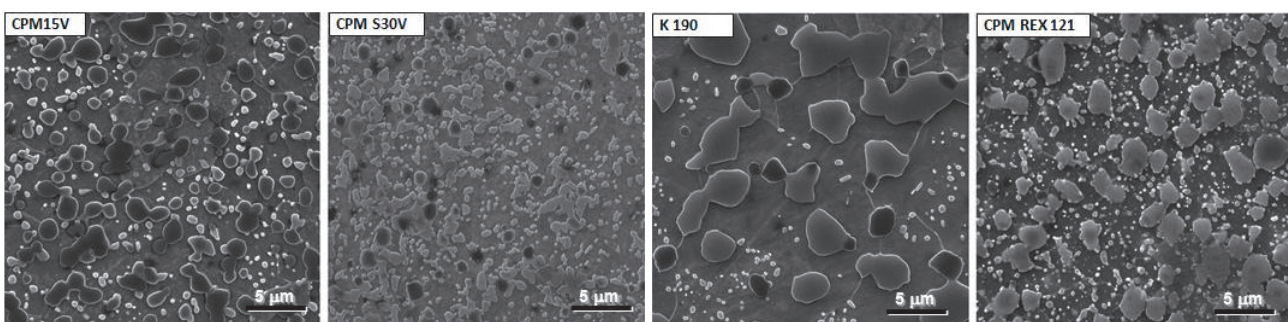


Fig. 1 Microstructures of steels used for experimental semi-solid processing

3. CHARACTERIZATION OF MICROSTRUCTURE PRIOR TO SEMI-SOLID PROCESSING

In the CPM 15V steel, the initial microstructure comprised ferritic matrix and globular vanadium and chromium carbides, **Fig. 1**. The phase composition was measured by means of diffraction. The matrix contained 72 % austenite, 18 % vanadium carbides (V₈C₇) and 10 % chromium carbides M₂₃C₆. The initial microstructure of CPM S30V was similar to this composition. According to X-ray diffraction analysis, it contained 70 % ferritic

matrix and balance of Cr₂₃C₆. It also contained a small amount of vanadium carbides which was, however, undetectable by diffraction.

The initial microstructure of K190 also comprised ferritic matrix making up 70 % of the material. Chromium carbides in the volume fraction of almost 30 % were balance of the composition. As in the previous case, there was a small amount of vanadium carbides. They were identified as the Cr₂₃M₆ type.

The microstructure of CPM REX 121 consisted of ferritic matrix with a high proportion of cobalt and carbides with bimodal size distribution. The larger carbides with an average size of 3 μm were complex vanadium-tungsten-molybdenum carbides. The smaller ones with sizes below 1 μm were M₂₃C₆-type chromium carbides.

4. MINI-THIXOFORMING

Semi-solid processing was performed in a specially-designed mini-thixoforming die. The feedstock with a cylindrical body of 48 mm in length and 6 mm in diameter had cone frustum ends to allow clamping between copper electrodes. It was heated inside the die by electrical induction and resistance. First, suitable forming temperatures had to be found for all materials. Their tentative values were first calculated (**Fig. 2**) [9]. The appropriate liquid fraction for thixo-forming is reported to be 10 - 30 % [10]. In terms of the temperature field control, the CPM REX 121 steel appears to best fit this range, as the desired liquid fraction is achieved at 1195 - 1220 °C. In the CPM S 30V, the required liquid fraction range lies between 1230 and 1250 °C. In K 190, the forming process window is somewhat narrower: 1235 - 1250 °C. The most difficult case in terms of process control is the CPM 15V steel. First, its forming process window is at higher temperatures, starting no lower than 1300 °C and, second, it is very narrow, causing the liquid fraction to increase very rapidly. Upon a temperature increase of a mere 18 °C, the material contains as much as 90 % melt. The temperature window for successful forming is thus approximately 5 °C. This turns the development of a new forming process into a considerable challenge.

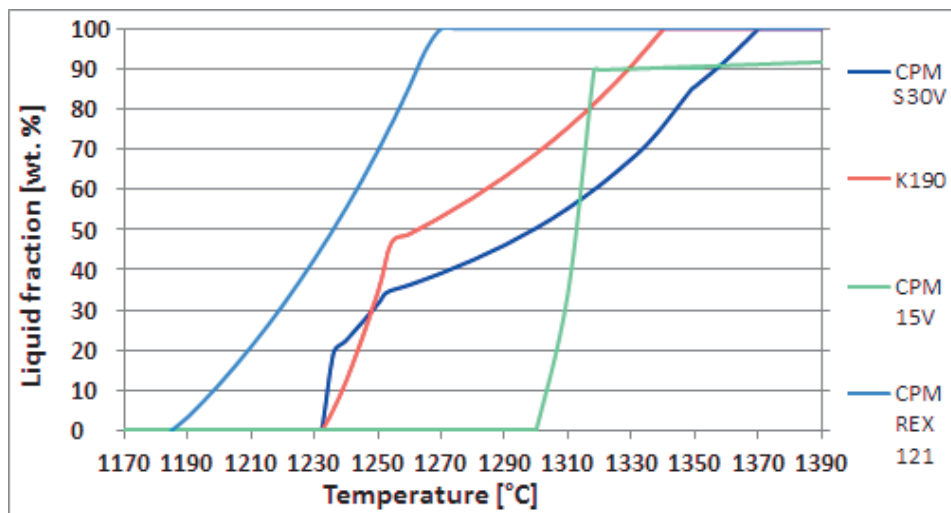


Fig. 2 Calculated melting curves of experimental materials

5. DISCUSSION OF RESULTS

5.1 Process Parameters

Phenomenological calculations provided a basis for trials with various heating temperatures for reaching the semi-solid state. It was found by experiment that theoretical computation needs to be verified and corrected

by trials. The process range was thus verified experimentally for each material. The first trial temperature for reaching the semi-solid state in the CPM 15V material was set at 1305 °C. At this level, the liquid fraction should be approximately 30 %. The total of heating and soaking times was 56 seconds. The specimen, however, melted in the process, indicating that the temperature was too high. Each next trial temperature was therefore 5°C lower than the last. The small step was chosen due to the very narrow temperature interval for achieving the semi-solid state. The heating temperature was therefore decreased in steps down to 1265 °C. After these heating trials with zero axial stress, schedules with pre-defined amounts of compressive deformation were tested as well. Based on deformation forces, the optimum forming range for the CPM steel was found to be 1265 - 1270 °C.

Parameters for the other steels were found in an equivalent manner. In the CPM S30V steel, the agreement between the calculated and experimental values was better. The heating trial was conducted using the temperature range of 1260 - 1280 °C. Upon heating at the upper limit temperature, metallographic observation revealed undesirable partially dendritic structure. The optimum temperature range was therefore narrowed to 1265 - 1270 °C .

As in the previous cases, the K190 was heated to the thixo-forming temperature for 56 s. The forming temperature was 1260 °C. Using these parameters, the desired shape was not obtained and other findings confirmed that the heating temperature chosen was too high. The liquid metal even entered the narrow parting plane gap in the mould. The high liquid fraction also caused liquid metal to be exuded in the end part of the mould. The heating temperature was thus reduced by 5 °C to 1255 °C. However, this heating temperature still proved to be too high. The molten material leaked out of the mould cavity along the longitudinal axis of the product, failing to fill the groove in the mould. The length of the resulting product was a mere 7 mm. By reducing the temperature in steps to 1240 °C, a high-quality product was eventually obtained. The metal then filled the mould cavity completely.

The first processing temperature chosen for trials on the CPM REX 121 steel was 1215 °C. The outcome showed that at this temperature, the liquid fraction required for adequate fluidity was not reached. By raising the heating temperature in steps of 5 °C, the liquid fraction was increased. Eventually, the entire mould cavity was filled and a product with the desired surface quality was obtained. The mould cavity was only filled completely at the temperature of 1235 °C.

5.2 Final Microstructure

Microstructures within the entire volume of the mini-thixoformed products were first examined using optical and scanning electron microscopes. The phase composition was measured by diffraction. The thixo-formed CPM 15V contained globular vanadium carbides embedded in austenitic matrix (**Fig. 3**). The matrix was first assumed to consist of a single phase. However, electron microscope observation confirmed that the matrix is an austenitic-martensitic mixture. This is in agreement with results of diffraction phase analysis. The resulting microstructure contained 50 % austenite and 30 % dispersed martensite. High-stability V8C7 carbides resisted melting during semi-solid processing and made up 20 % of the structure. Carbides with greater chromium content melted completely and their elements formed a eutectic, predominantly on grain boundaries.

Similar microstructure was found in the CPM S30V steel. The final microstructure of this steel consisted of 70 % of austenite, 14 % martensite and 16 % Cr23C6 carbides (**Fig. 3**). The high amount of austenite was retained thanks to chromium carbides which partially melted during semi-solid processing and some chromium migrated to the austenitic matrix. The remaining carbides reprecipitated in the form of network, predominantly on grain boundaries.

The resulting microstructure of K190 steel was similar to that of the other materials. M-A constituent prevailed and its grains were surrounded by carbide network. Diffraction phase analysis revealed an increased

proportion of martensite at the expense of austenite. The martensite and austenite volume fractions were 19 % and 59 %, respectively. The balance constituent consisted of Cr23M6 carbides.

The final microstructure of the CPM REX 121 steel consisted of austenitic matrix and a small fraction of martensite needles. Primary chromium carbides with sizes below 1 µm partially melted and transformed into a eutectic located at austenite grain boundaries. Their volume fraction increased slightly with the processing temperature. Complex V-W-Mo carbides, on the other hand, retained their initial character. In some cases, multiple carbides coalesced into larger particles. Isolated carbides became more globular than in the initial condition.

5.3 Mechanical Properties

As the powder materials in question exhibit very low tensile strength and virtually zero elongation, these parameters are neither measured nor listed in their data sheets. These materials are normally

evaluated on the basis of their hardness and wear-resistance. Hardness was thus chosen as the reference parameter to compare their mechanical properties before and after thixo-forming. The CPM 15V steel in its initial condition showed the average hardness of 298 HV10. Upon thixo-forming, its hardness was 728 HV10, which is a 2.5-times higher value. This increase can be attributed mainly to the formation of martensite within the matrix and to the chromium precipitates in the form of network.

The hardness of the initial CPM S30V steel (310 HV10) was somewhat higher than that of the CPM 15V steel. Semi-solid processing did not lead to any significant increase in hardness, the final value of which was 468 HV10. The relatively low strength when compared to the other materials was probably due to the high volume fraction of austenite in the microstructure.

K190 showed the lowest initial hardness of all experimental materials: 232 HV10. As the chemical composition of this steel is similar to that of CPM S30V and its carbon content is even higher than that of CPM S30V, the markedly lower yield strength can be attributed to the substantially coarser carbides in K190. However, its hardness increased greatly upon semi-solid processing, reaching 543 HV10. This was probably owing to the high carbon content which caused a high volume fraction of martensite to form within the austenitic matrix.

By contrast, CPM REX 121 showed the highest initial strength, 407 HV10, of all materials. It had a high carbon content and, consequently, high volume fraction of martensite upon processing. This led to a notable increase in hardness to 830 HV10, which is the highest final value among all the experimental materials. A contribution

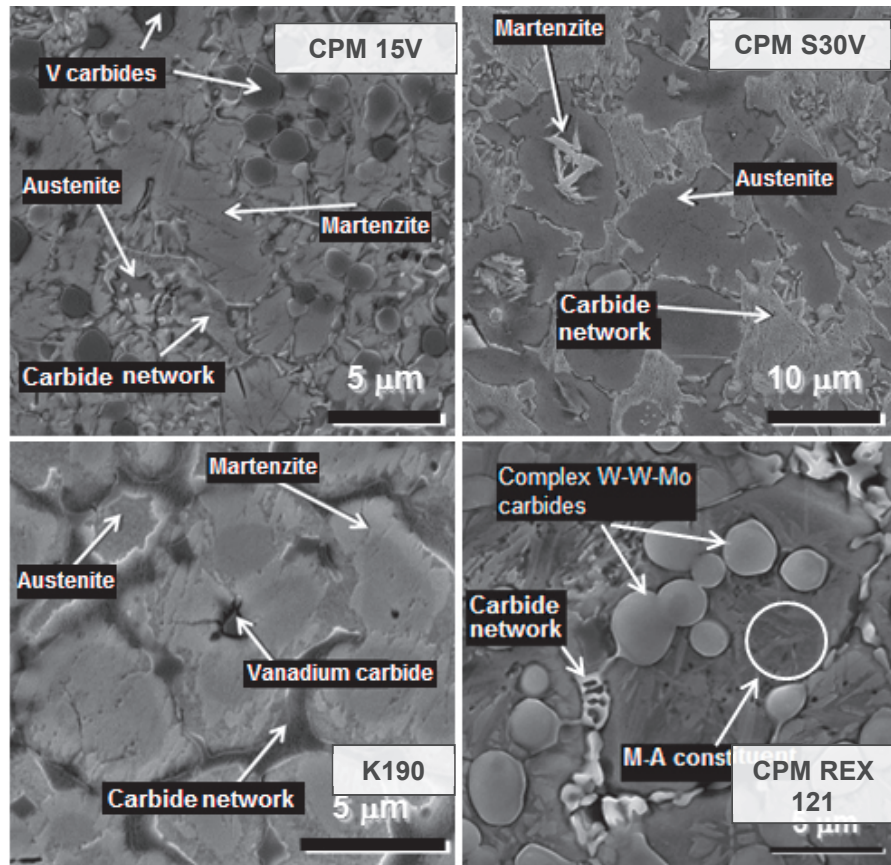


Fig. 3 Resulting microstructures after mini-thixoforming

to this increase can be attributed to high levels of alloying elements, and thus a high proportion of undissolved carbides in the microstructure.

CONCLUSION

In the present experiment, CPM 15V, CPM S30V, K 190 and CPM REX 121 steels were processed by mini-thixoforming, which is a promising technique for their processing. Mini-thixoforming is a method for the manufacture of highly complex-shaped parts with high hardness and wear resistance even under extremely harsh application conditions. The temperature intervals for processing these steels are, however, very narrow when compared to the materials typically used. As a result, the process is difficult to control and poses a considerable technological challenge. In order to successfully produce complex-shaped demonstration products with dimensions on the order of millimetres, exact processing parameters and their maximum permissible deviations must be found. For this purpose, tentative temperatures for thixo-forming were first calculated for all materials using phenomenological models. Then they were corrected according to experimental results and the optimum processing temperatures were determined. Once the forming temperatures were optimized, other processing parameters were refined as well in order for the material to completely fill the mould cavity. In all cases, the resulting microstructure upon forming consisted of polyhedral austenite grains with various levels of martensite. These grains were embedded in ledeburite network. With increasing carbon content, the volume fraction of martensite upon mini-thixoforming increases. This, in addition to the presence of carbides, has a major impact on the increase in hardness. This increase was most apparent in the CPM 15V and CPM REX 121 materials. The hardness level achieved in the CPM 15V steel was 728 HV10. In the CPM REX 121 steel it was as high as 830 HV10.

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REFERENCES

- [1] PÜTTGEN, W., BLECK, W., HIRT, G., SHIMARA, A. Thixoforming of Steels - A Status Report. *Advanced Eng. Mater.*, 9, 4, 2007, 231-245.
- [2] KIRKWOOD, D.H., SUÉRY, M., KAPRANOS, P., ATKINSON, H.V., YOUNG, K.P. *Semi-solid metal processing*, 2010, XII, 172 p.
- [3] SEN, I., JIRKOVA, H.; MASEK, B., BÖHME, M., WAGNER, M. F. Microstructure and mechanical behavior of a mini-thixoformed tool steel. *Metal. Mater. Trans.*, 43, 2012, 3034-3038.
- [4] MASEK, B. et al. Structure of Miniature Components from Steel Produced by Forming in Semi-solid State. *Trans. Nonferrous Met. Soc. China.*, 20, 2010, 1037-1041.
- [5] JIRKOVÁ, H., AIŠMAN, D., SEN, I., WAGNER, M. F.-X., BEHULOVA, M., KUSY, M., MASEK, B. Mini-thixoforming of a Steel Produced by Powder Metallurgy. *Solid State Phen.*, 192-193, 2013, 500-505.
- [6] ELIÁŠOVÁ, I., MAŠEK, B., KUSÝ, M., SVOBODA, J., JIRKOVÁ, H. The Influence of the Temperature of the Processing on the Final Structure of the Metal-Ceramic Compact Obtained through the Semi-Solid state. In: *Metal 2012*, Ed. TANGER s.r.o. Ostrava, 2012.
- [7] VANČURA, F., MAŠEK, B., AIŠMAN, D., JIRKOVÁ, H., WAGNER, M.F.-X., BÖHME, M., Modification of metastase microstruktura of CPM15V steel by heat exposure after treatment in semi-solid state. *J. Alloys Counds*, 586, 2014, 159-164.
- [8] MAŠEK, B., AIŠMAN, D., JIRKOVÁ, H., KUSÝ, J. Development of Microstructure from Powder Tool Steel Obtained through Semi-solid Metal Working, M. in: *Metal 2012*, Ed. TANGER s.r.o. Ostrava, 2012.
- [9] *JMatPro*, The Java-based Materials Property Simulation Package, Version 6.1, Sente Software Ltd., Surrey Technology Center, UK.