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Mini-thixoforming of a Steel Produced by Powder Metallurgy

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Abstract. Semi-solid processing is complicated by various inherent technical problems. However, once these problems are solved, thixoforming allows intricately shaped components to be manufactured very effectively – often with microstructures that cannot be produced by any other techniques. The recently introduced mini-thixoforming method is an example of such a novel technique for semi-solid processing of steel. The wall thicknesses of resulting parts are about 1 mm. Microstructures of semi-solid-processed steels typically consist of a high proportion of globular particles of metastable austenite embedded in a carbide network, the latter being much harder and more brittle. This paper illustrates that mini-thixoforming allows inverting that microstructural configuration. As an experimental material, powder steel with increased content of vanadium and chromium was used. The post-thixoforming microstructure consisted of a dispersion of carbides and high-vanadium and high-chromium eutectic in an austenitic matrix. Applying optimised processing parameters, complex-shaped parts could be produced. According to the high hardness of resulting microstructural components, the new materials are likely to exhibit extraordinary strength and wear resistance.

Introduction

Thixoforming of steels represents a technological challenge due to the requirement for high processing temperatures [1]. Although the thixoforming technique of low-melting-point materials were invented as early as 1972 [2], it took nearly twenty years to develop the technology for steels [1]. Apart from economic considerations, introduction of thixoforming of steel to industry is driven by other factors. Thixoforming simplifies forming processes, enables intricately shaped steel parts to be produced effectively, enhances productivity and offers the advantages of near net shape forming [1].

In selecting a steel for the process, great emphasis must be laid on its thixoformability which depends on the temperature range between solidus and liquidus temperatures of the steel , on the suitability of its semi-solid condition for forming and on further factors, see reference [1]. For steels with a wider freezing range and, at the same time, lower liquidus and solidus temperatures, the process is more reliable and economically beneficial [3]. The thixotropic properties of a material considerably depend also on the proportion of the material in the liquid state [1, 4-5].

Typically, the microstructures of semi-solid-processed tool steels consist of metastable austenite and a fine eutectic network [6]. This is, e. g., commonly observed in the X210Cr12 steel, where the globular particles and eutectic network exhibit hardness values of about 300-350 HV0.05 and 500-600 HV0.05, respectively [6-7]. If this microstructural configuration could be inverted, i.e., if hard particles were embedded in a ductile matrix, various mechanical properties (such as wear resistance) could likely be considerably improved. This paper describes how semi-solid processing of a crucible particle metallurgy (CPM) tool steel with higher vanadium content, CPM 15V, can be used to produce such an inverted microstructure.

Experimental Programme

Experimental material. In order to alter the microstructure as described above, a non-standard thixoforming material had to be used. The powder steel CPM 15V with the chemical composition given in Table 1 was selected for the experiment. CPM 15V is a high-vanadium and high-chromium steel with a carbon content of 3.4 %, intended for production of tools with extraordinary wear resistance and high hardness [8], such as tools for drawing, shearing, cutting and similar applications.

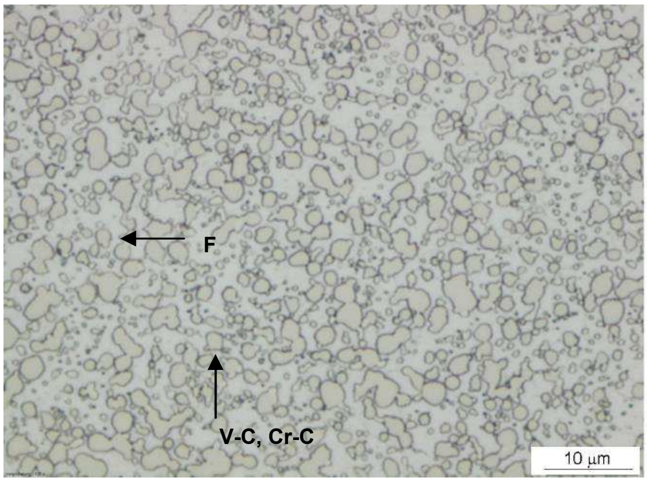


Fig. 1 Initial microstructure of CPM 15V

The microstructure of the steel in as-received state consisted of a ferrite matrix and uniformly distributed chromium-vanadium carbides with a size of ~1.6 μm, corresponding to the volume fraction of approximately 0.34 (Fig. 1). Calculations carried out in the JMatPro software [9] revealed that the freezing range for the investigated steel is 350 °C but the temperature interval available for mini-thixoforming corresponding to the volume portion of liquid phase from 0.4 - 0.6 is only 15 °C.

Table 1 Chemical composition of the CPM 15V steel

C	Cr	V	Mo	Mn	Si
3.40	5.25	14.5	1.30	0.50	0.90

Mini-thixoforming. Semi-solid processing was performed in a specially designed mini-thixoforming titanium die [10]. The feedstock with a cylindrical dimension of 42 mm in length and 6 mm in diameter had blunt cone ends to allow clamping between copper electrodes. The pieces were heated in the die cavity by combined electrical induction and resistance methods. The feedstock was heated to 1270 °C over 56 seconds and then compressed to a height of 9 mm. The semi-solid material was thus forced to flow from the die into the mould cavity. Several different moulds were used to obtain various product shapes, including a straight, curved and narrow straight channels and a pyramid frustum (Fig. 2).

The process of material flow and solidification in the mould cavity was examined using optical and scanning electron microscopes on metallographic sections through the products. Hardness was measured in multiple locations of the products. Volume fractions of microstructure components were identified using X-ray diffraction analysis in a diffractometer with Co-Kα source.

Results and Discussion

Processing in semi-solid state. Various shapes of the product mould were used for optimising key process parameters, namely the heating temperature and level of reduction. For the initial tests of the minithixoforming of the CPM 15V steel, a cuboid mould cavity with a straight section of 5×3 mm^2 and a length of 15 mm was used. The entire mould cavity was filled and a high-quality surface was obtained (Fig. 2a). Fig. 2b shows a miniature tension test specimen that was formed in a single process step. The size of the mould cavity included allowances for polishing the specimen prior to testing. Heating temperature and other parameters were the same as with the previous mould shape. The length of the cavity was 20 mm; the gauge section width was 2 mm, and the width of the specimen head was 5 mm. The thickness of the specimen was a mere 1.9 mm. Upon surface inspection of the product it was found that the wide end of the cavity was filled by the material flowing back after the contact with the mould front face. Another mould with a 2.3×1.9 mm^2 section was used to explore the fluidity of the material, i.e., its ability to fill a thin curved channel. The mould was filled up to 21 mm depth (Fig. 2c). The ability of the metal to fill a pyramid frustum cavity was tested using a channel with initial and final dimensions of 2.0×1.5 mm^2 and 1.8×1.5 mm^2 respectively (Fig. 2d). A mould with ECAP channel cavity (L-shaped) was used to examine the ability of the material to fill cavities with a 90° bend. The length of the channel was 13 mm, its cross-section through the bend 4×1.9 mm^2. The end was made narrower, forming a pyramid frustum to cause counter pressure while the material is flowing around the bend in the cavity.

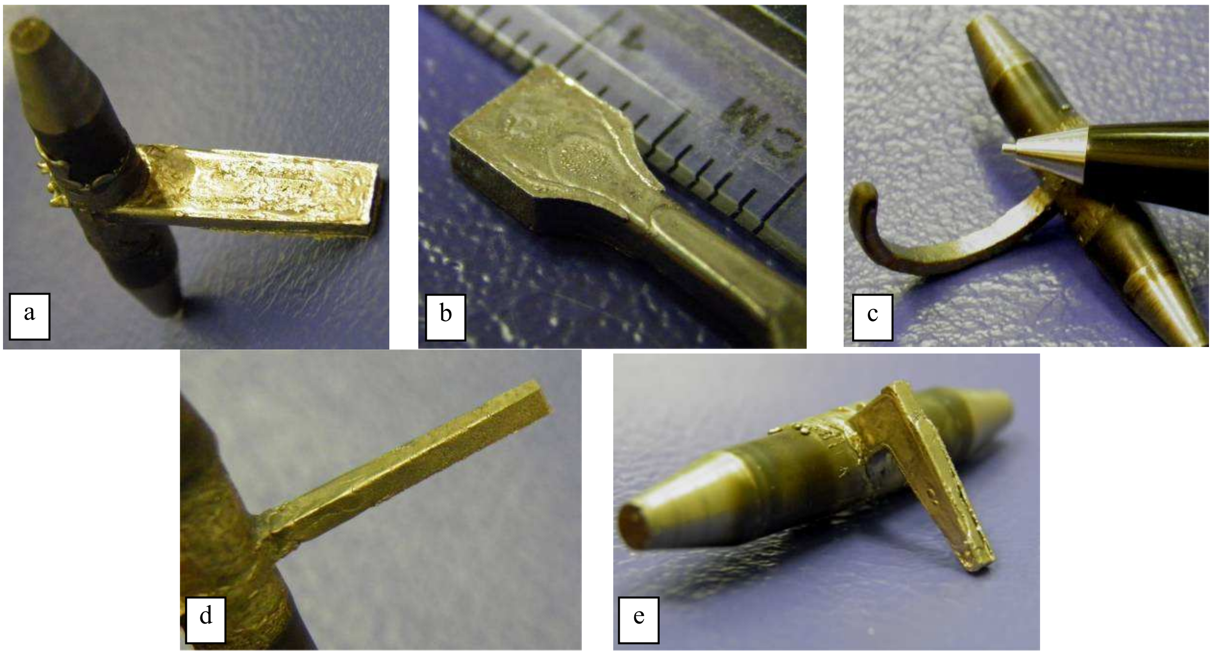
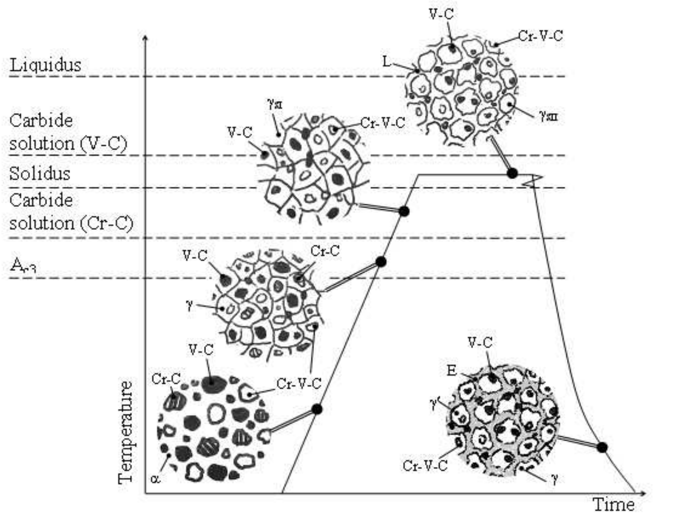


Fig. 2 Shapes of products produced by mini-thixoforming from the CPM 15V steel: a) cuboid, b) miniature tension test specimen, c) curved cuboid d) pyramid frustum, e) ECAP channel shape (L-shape)

Analysis of resulting microstructure. As expected, semi-solid processing altered the microstructure of the material considerably. The chemical composition of the material, high heating rate and rapid cooling caused the initial ferrite matrix to transform into an austenite-martensite matrix with increased Si content. The majority of chromium carbides were dissolved during the heating. During solidification, a chromium-vanadium eutectic developed. In the structure, vanadium carbides with a melting point of 1280 °C remained undissolved (Fig. 3).

The morphologies and distributions of the different phases vary with cooling and solidification conditions. This is shown very clearly in an overview image of the cross-section from a curved part in



α – Ferrite	V-C – Vanadium carbides
γ – Austenite	Cr-C – Chromium carbides
γ' – Martensite	Cr-V-C – Chromium-vanadium carbides
γ_{SI} – Supersaturated austenite in solid state	E – Eutectic
γ_{SII} – Supersaturated austenite in semi-solid state	L – Liquidus

Fig. 3 Structure development during semi-solid state processing

Fig. 4. The central part at the mould inlet consists of a coarse eutectic network, parts of which lie on austenite grain boundaries (Fig. 4b). A coarse microstructure can be observed along the entire longitudinal axis of the product (Fig. 4c). The phases are distributed more evenly near the surface of the product (Fig. 4d), most likely due to a higher solidification rate. Complementary Finite Element calculations [10] indicate that the cooling rates are relatively high and the surface of the product is cooled down below the equilibrium solidus temperature during a time of about 0.3 s. The liquid phase remains only in the central region [10].

Consequently, the central regions contain larger and coarser eutectic microstructures. The feedstock contains less eutectics than the product. This is due to the fact that more liquid phase was pushed out during compressive deformation into the mould cavity in which it rapidly solidified in non-equilibrium conditions. Shrinkage cavities are present along the longitudinal axis of the feedstock (macrograph in Fig. 4). They were also found at the ends of products. The cavities were smaller than a few micrometers and typically arranged in clusters. There were no significant differences between products made in the different moulds (Fig. 5 – Fig. 7).

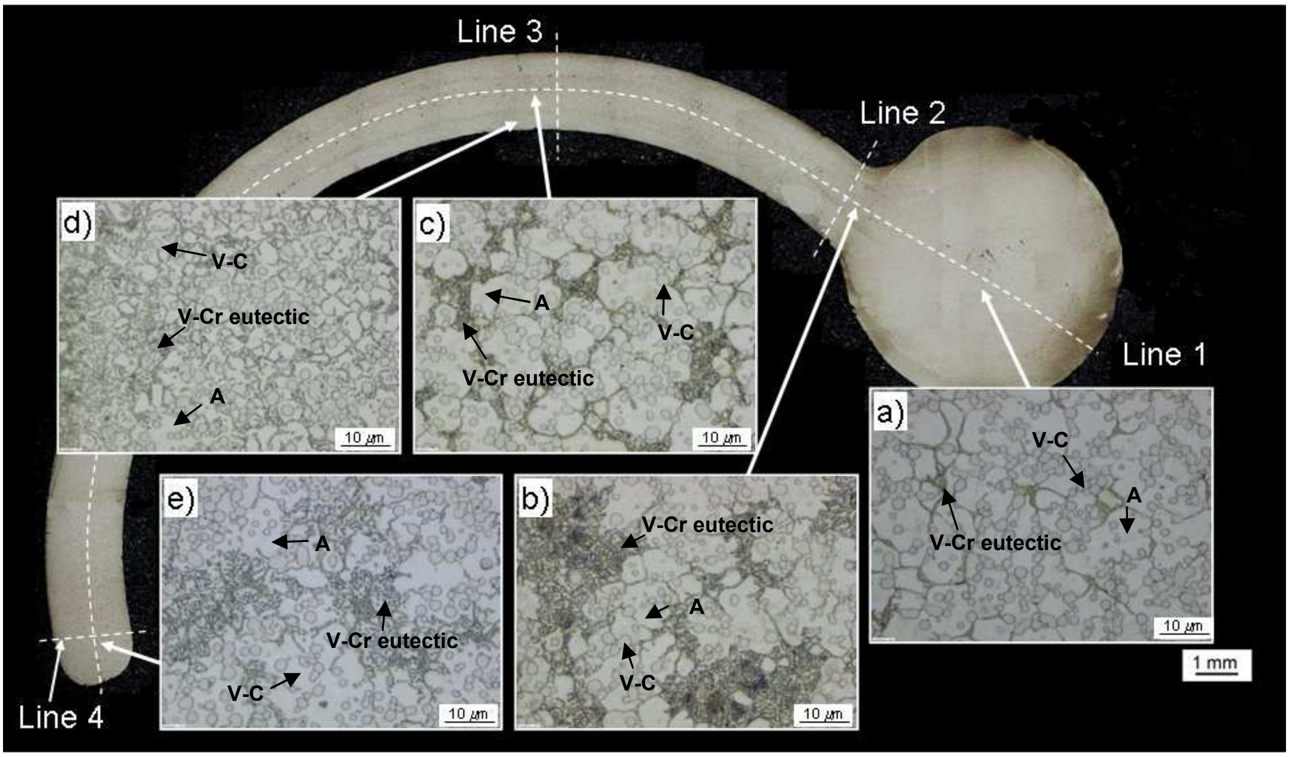


Fig. 4 Macrograph of a section through the curved product with detailed micrographs: (a) feedstock centre, b) mould inlet part, c) centre of product, d) surface layer of product, e) centre of the tip of product

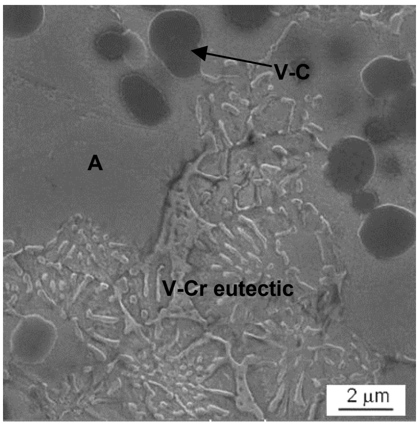


Fig. 5 Centre of the curved product

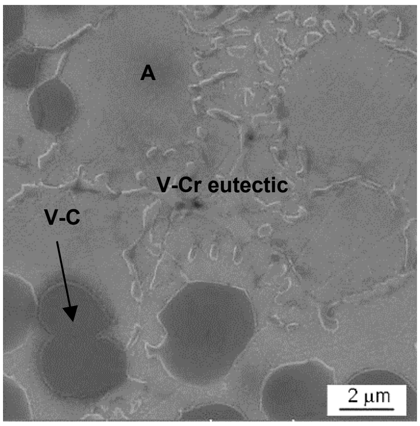


Fig. 6 Centre of the gauge section of the tension test specimen

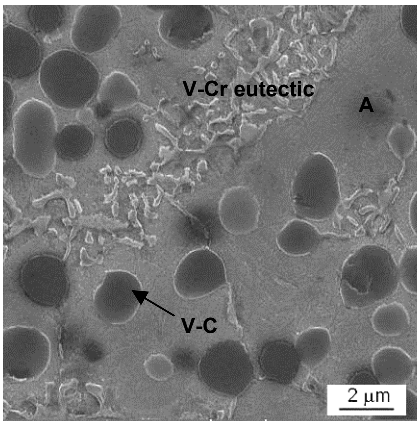


Fig. 7 Centre of the pyramid frustum product

Hardness was measured along the longitudinal axis of the products, as well as along three transverse lines (see Fig. 4 – lines 1-4). A load of 5 kg was used for the hardness measurements. The results are shown in Figures 8 and 9. Products of all shapes showed similar hardness values of about 783 HV5. Hardness along their longitudinal axes (line 1) decreases near the product tip (Fig. 8). A notable difference between the hardness of the product body and that of its tip was found in the ECAP (L-shaped) product: 792 HV5 and 672 HV5, respectively. Variations were found in the curved product as well: the hardness near the mould inlet was 792 HV5; the central part of the product exhibited 672 HV5, whereas the hardness of its tip was 762 HV5. Along the profiles (lines 2-4) closer to the product tip, the lower hardness was measured. The presence of the coarse eutectic phase in the product centre does not appear to affect the hardness values. The hardness near the mould inlet (line 2) was about 780 HV5. In the centre of the product (line 3), it was lower (700 HV5), whereas near the product tip (line 4), it was about 660 HV5 (Fig. 9). Again, in the curved product, hardness along line 3 was lower than that along lines 2 and 4 (Fig. 9).

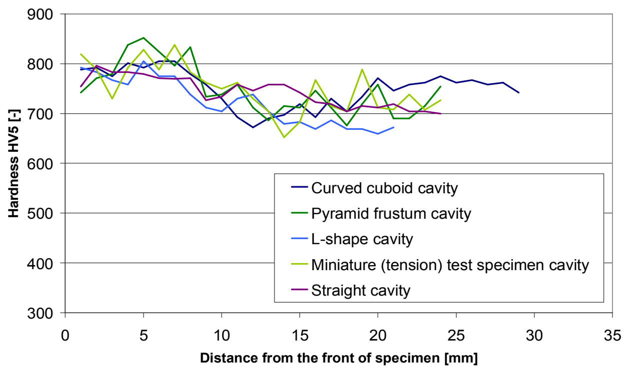


Fig. 8 Hardness profile along line 1 on the product axis

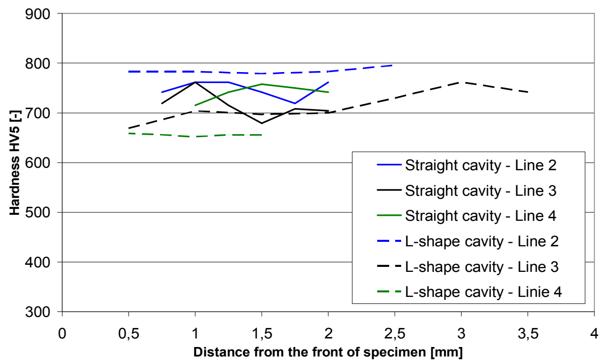


Fig. 9 Hardness profiles along lines 2, 3, 4 in curved and L-shaped (ECAP shape) products

Table 2 Fraction of individual phases on the product beginning and tip (X-ray diffraction analysis)

Form	Position on the product	V-C [%]	Martensite [%]	Austenite [%]
Cuboid	front	14	24	62
	tip	17	20	63
Curved	front	13	35	53
	tip	15	49	35
L-shape	front	15	30	55
	tip	20	24	56
Tension test specimen	front	14	38	49
	tip	17	37	44
Pyramid frustum	front	14	45	41
	tip	10	59	31

The volume fractions of individual phases in the product near the mould inlet and in the tip were measured using XRD (Table 2). The largest austenite fraction in the structure was detected in the cuboid product with the constant cross section along the whole length. In the front part of the product (line 2) as well as at the tip (line 4), approx. 62 % of austenite was obtained. The fraction of the V-C carbide oscillated around 14-16 %. A significant increase of the martensite fraction was observed at the tip of the products with very narrow cross sections. In the curved and the pyramid frustum products, the martensite fractions increased from 35 up to 49 % and from 45 to 59 %, respectively (Table 2). This increase was caused by the higher cooling rate at the location of the narrow cross section. The 90° bend led to a slight increase of V-C carbides from 15 to 20 % in the L-shaped product.

Summary and Conclusions

Mini-thixoforming was used to produce demonstration products of various shapes from a CPM 15V powder metallurgy steel. An inversed configuration of the microstructure was developed during mini-thixoforming compared to conventional microstructures found in thixoformed steels: the mini-thixoformed microstructure consists of hard carbides and of chromium-vanadium eutectic, both embedded in the austenitic-martensitic matrix. Using optimised forming conditions, demonstration products of various shapes could be produced in a titanium mould. Among them were products with a thickness of a mere 1.8 mm. A mould cavity with a cross-section of 2.3×1.9 mm^2 was filled successfully up to a depth of 21 mm, as well as a cavity with a 90° sharp bend. Moreover, a miniature tensile test specimen was successfully produced in a single step. This will allow the mechanical properties of semi-solid-processed materials to be determined directly in future investigations. The obtained results demonstrate how broad the hidden potential of unconventional semi-solid processing still is. Surprising possibilities open up in terms of novel microstructures in a traditional material that has been optimised for millennia: steel.

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