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Development of microstructure in high-alloy steel K390 using semi-solid forming

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Abstract. Semi-solid processing of light alloys, namely aluminium and magnesium alloys, is a widely known and well-established process. By contrast, processing of powder steels which have high levels of alloying elements is a rather new subject of research. Thixoforming of high-alloy steels entails a number of technical difficulties. If these are overcome, the method can offer a variety of benefits. First of all, the final product shape and the desired mechanical properties can be obtained using a single forming operation. Semi-solid forming can produce unusual powder steel microstructures unattainable by any other route. Generally, the microstructures, which are normally found in thixoformed steels, consist of large fractions of globular or polygonal particles of metastable austenite embedded in a carbide network. An example is the X210Cr12 steel which is often used for semi-solid processing experiments. A disadvantage of the normal microstructure configuration is the brittleness of the carbide network, in which cracks initiate and propagate, causing low energy fractures. However, there is a newly-developed mini-thixoforming route which produces microstructures with an inverted configuration. Here, the material chosen for this purpose was K390 steel, in which the content of alloying elements is up to 24 %. Its microstructure which was obtained by mini-thixoforming did not contain polyhedral austenite grains but hard carbides embedded in a ductile austenitic matrix. This provided the material with improved toughness. The spaces between the austenite grains were filled with a eutectic in which chromium, molybdenum and cobalt were distributed uniformly. After the processing parameters were optimized, complex-shaped demonstration products were manufactured by this route. These products showed an extraordinary compressive strength and high wear resistance, thanks to the hardness of their microstructure constituents, predominantly the carbides.

Keywords: Semi-solid state, high-alloy steel, K390, thixoforming

1. Introduction

Semi-solid processing of powder steels is a new and technically interesting technique which enables complex components with advanced properties to be produced using a single forming operation. It allows engineers to combine the advantages of complex-shapes of castings and mechanical properties of forgings in materials which – due to their high alloy levels – are difficult to form by conventional methods, not to mention making small and intricate-shaped parts by forming [1]. The purpose of the present experiment was to explore the potential of the above-mentioned material for mini-thixoforming. The process parameters were gradually optimized in order to obtain complex-shaped products. The forming process took place in a closed cavity of a die which had been specially



developed for the mini-thixoforming technique. Using this tool, thin-walled products with the wall thickness of approximately 1 mm can be made.

Thixoforming involves heating a material to the region between the solidus and liquidus temperatures and forming it in a partially melted state [2]. Relevant data for this experiment was predicted with the aid of calculation in JMatPro software. Based on this data, initial conditions were specified for this steel. The most important of those was the temperature, at which the material contains 15 % liquid fraction. The preliminary calculation clearly showed that achieving the desired liquid fraction would be difficult. The reason is that this parameter increases very steeply with temperature. The temperature range available for semi-solid forming thus becomes narrow: approximately 60 °C (Fig. 1). For this reason, the process parameters were subsequently modified, namely the heating profile and temperature.

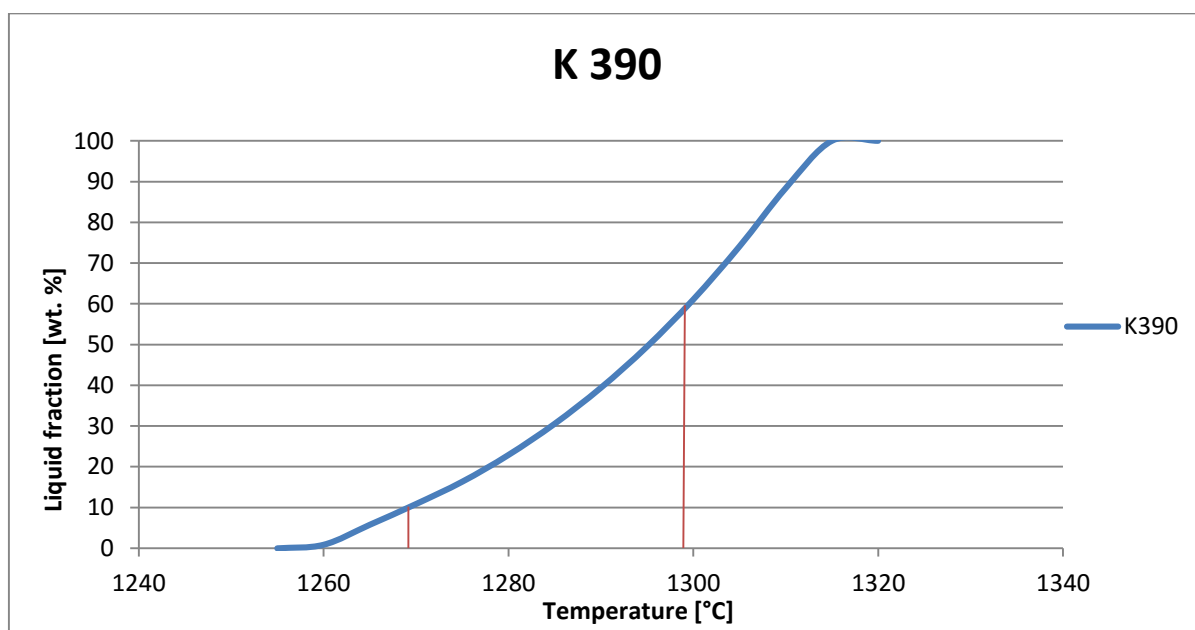


Figure 1. The actual temperature interval for effective thixoforming – liquid between 10 % and 60 %.

2. Experimental programme

In order to obtain the desired “inverted” microstructure, the chemical composition of the material had to be altered adequately, as it was substantially different from the chemistries of those steels which are normally used for thixoforming experiments [3]. The objective was to preserve carbide particles in the material in semi-solid state. After the process, these would remain embedded in metastable austenite. The formation of carbide network would be suppressed. Based on calculations, the high-alloy K390 powder steel was chosen as the experimental material. It has high vanadium (up to 8.94 %) and carbon (up to 2.48 %) levels (Tab. 1). Given its high hardness and wear resistance, the K390 steel is typically used for making tools.

Its initial microstructure contains a ferrite matrix with high levels of, molybdenum and chromium, and with uniformly distributed carbides. Its hardness is 255 HV30. The carbides are mainly vanadium carbides with sizes between tenths of micrometres and 2 µm. Image analysis shows that their proportion in the microstructure is approx. 35 % (Fig. 2). Distributions of alloying elements were mapped using microscopic observation and EDS analysis (Fig. 3).

Table 1. Chemical composition of experimental material.

Amounts of alloying elements [wt. %]	C	Cr	Cu	Co	Mn	Mo	Ni	Si	V	W
K390	2.48	4.20	0.10	1.95	0.40	3.64	0.19	0.57	8.94	0.85

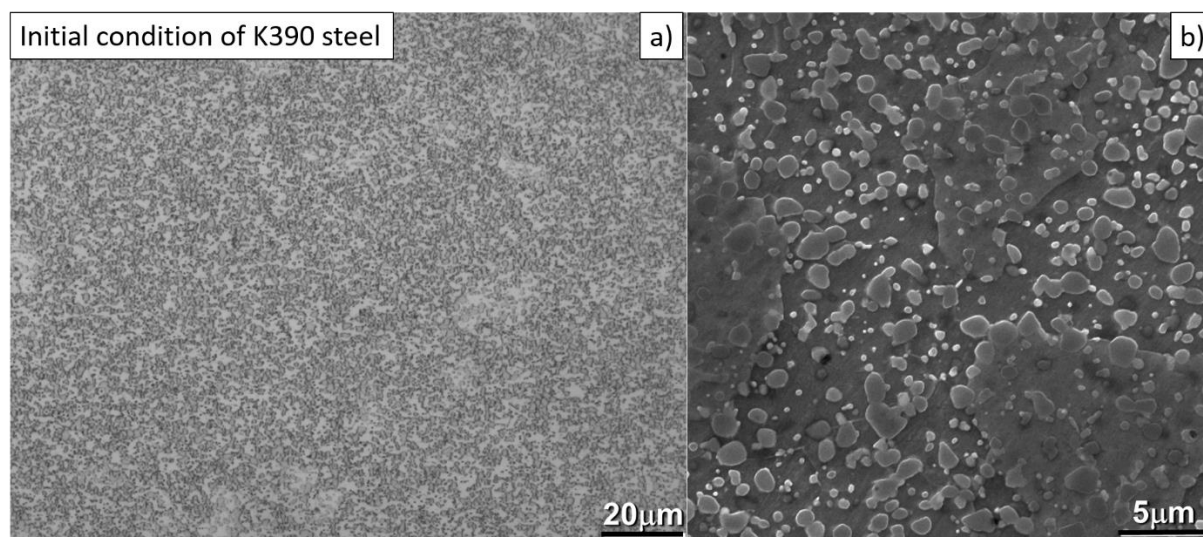


Figure 2. Microstructure of the material in its initial condition: a) light microscope image, b) SEM image.

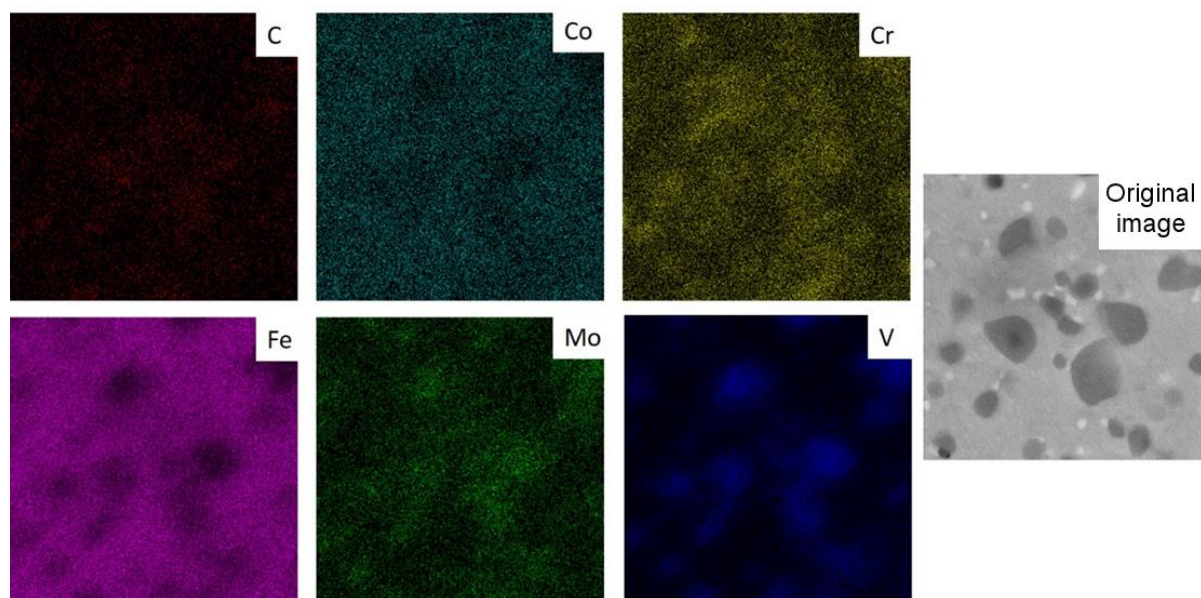


Figure 3. Maps of distributions of chemical elements in the initial condition of the material.

3. Choice of parameters and semi-solid processing

The experiment was carried out in a specially developed device which comprised a titanium die for mini-thixoforming. Cylindrical stock of 6 mm diameter and 50 mm length with truncated cone ends

was used. The purpose of the conical ends was to align the stock to the axis of the die and provide contact surfaces for energy supply via copper grips. In order to provide a wider range of product shapes, the die had an exchangeable shape insert. The stock was heated by electrical resistance and induction while placed in the die. The design of the forming device provides high rates of heating, solidification and cooling. With a digital high-frequency control system, the entire process could be controlled accurately, including the forming route and the heating profile [4].

As this process is impossible to describe and calculate to adequate accuracy using numerical methods, it was optimized iteratively by experiment. The forming process parameters were optimized, until the die cavity filled completely with semi-solid metal and the product's final microstructure was uniform. An important aspect of mini-thixoforming is accurate temperature control for achieving the desired liquid fraction. It was found that the K390 steel begins to melt at 1255 °C and becomes fully melted at 1315 °C. The available range for semi-solid forming is therefore approximately 60 °C. The actual temperature interval for effective thixoforming is, however, narrower because the liquid fraction should be kept between 10 % and 60 % [5].

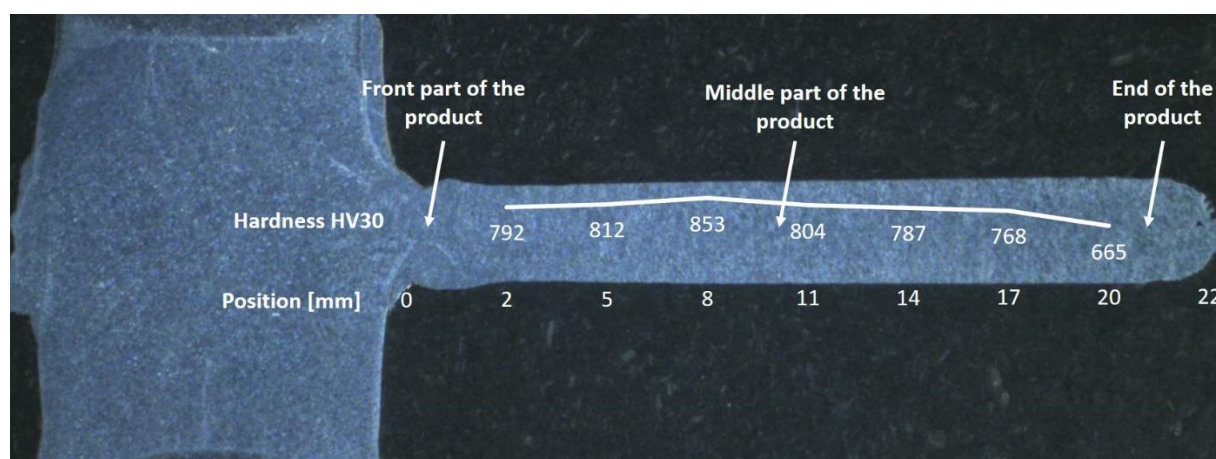


Figure 4. Hardness and microstructure analysis on axial cross-section through the product.

4. Results and discussion

The first forming run took place at a temperature at which, according to the preliminary calculation, the material should contain 15 % liquid fraction. The stock was heated to 1270 °C over 56 seconds and then formed using a maximum force of 6.5 kN. The deformation itself took less than 0.3 seconds. This temperature proved too high, as the stock lost shape stability owing to excessive melting. The material made uncontrolled local contact with the die wall, and the temperature field was thus disturbed. As a consequence, the die cavity was not filled. In the next runs, the temperature was reduced step-by-step, until the appropriate temperature for complete filling of the die cavity was found. In each case, the material flow through the die cavity was subsequently studied by metallographic observation of transverse sections through the product [6]. Besides optical microscopic and electron microscopic analysis, hardness measurement was undertaken in various locations within the product (Fig. 4). Amounts of individual microstructural constituents were determined by X-ray diffraction analysis (using a diffractometer with a Co-K α source).

Heating to 1253 °C was found to be the most suitable option. At this temperature, the stock becomes melted to a sufficient extent, while retaining shape stability. It is then easily extruded into the product die cavity.

Microstructures of the products were studied. In accordance with the expected use of high-stability carbides of vanadium, an “inverted” microstructure was produced. The chemical composition of the material, the high heating rate and rapid cooling caused the initial ferrite matrix to transform into an

austenite-martensite mixture. Chromium-vanadium eutectic formed as well, and the initial vanadium carbides were retained. The temperature at which the latter dissolve is 1280 °C. The processing temperature was thus lower enough than this dissolution temperature. These retained carbides were more spherical than their initial form, due to diffusion and mechanical effects, similar to the newly-formed ones [7]. The chromium-molybdenum-vanadium eutectic was present along austenite grain boundaries. Scanning electron microscopic observation and EDS studies, performed on Zeiss microscope with Oxford Instruments detectors, showed that a majority of alloying elements redistributed during thermomechanical processing and that the nature of the matrix changed profoundly (Fig. 5). The microstructure was homogeneous along the entire product axis. Only the region near the entry opening of the die cavity contained less globular and non-uniformly distributed carbides (Fig. 6). This was caused by the short path they travelled and by the less intensive mechanical interaction with the melt and other solid particles. These joint effects also slowed down diffusion which normally greatly contributes to spheroidization.

As the nature of the matrix changed and part of the super-saturated austenite transformed to martensite, the hardness increased to a level as high as 853 HV30.

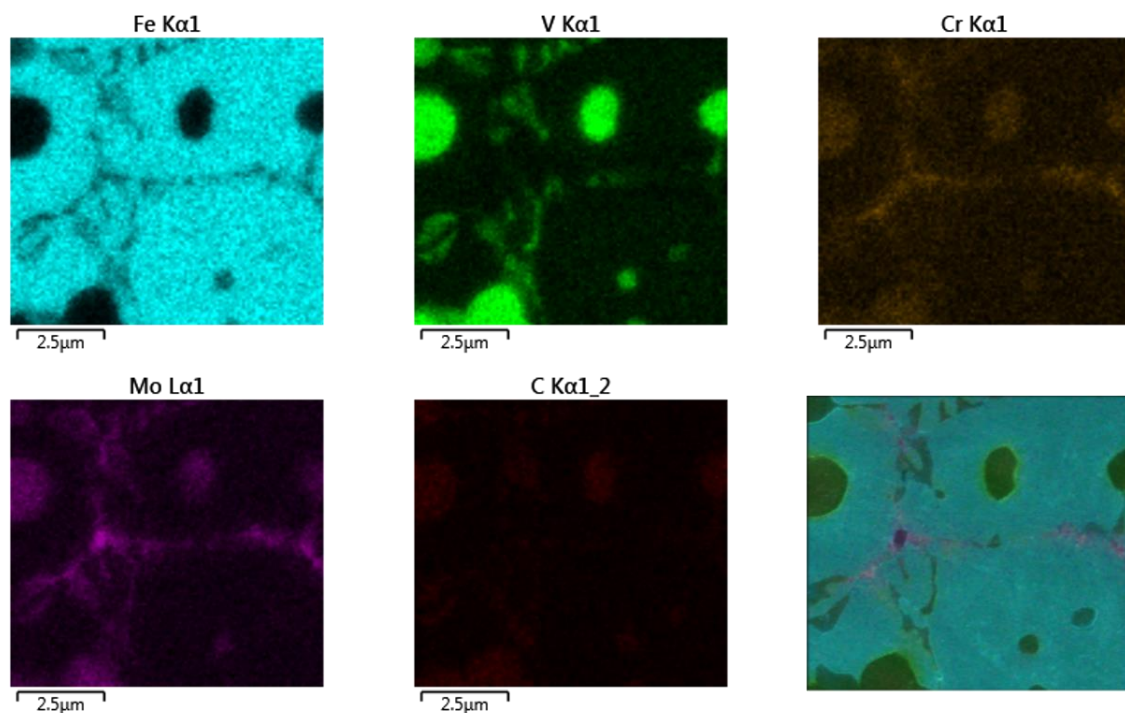


Figure 5. EDS Maps of redistributions of chemical elements after mini-thixoforming.

X-ray diffraction analysis was employed to determine the fractions of microstructural constituents in the product. The matrix consisted of austenite grains, carbide network along grain boundaries and a low fraction of martensite needles (Fig. 6 c)). The presence of martensite could also be derived from hardness values (Fig. 4). The amounts of individual phases in the microstructure of K390 steel are in agreement with its chemistry. Given the high amount of vanadium, a majority of carbides contained this element. The structure also includes chromium and molybdenum carbides which are less frequent (Tab. 2).

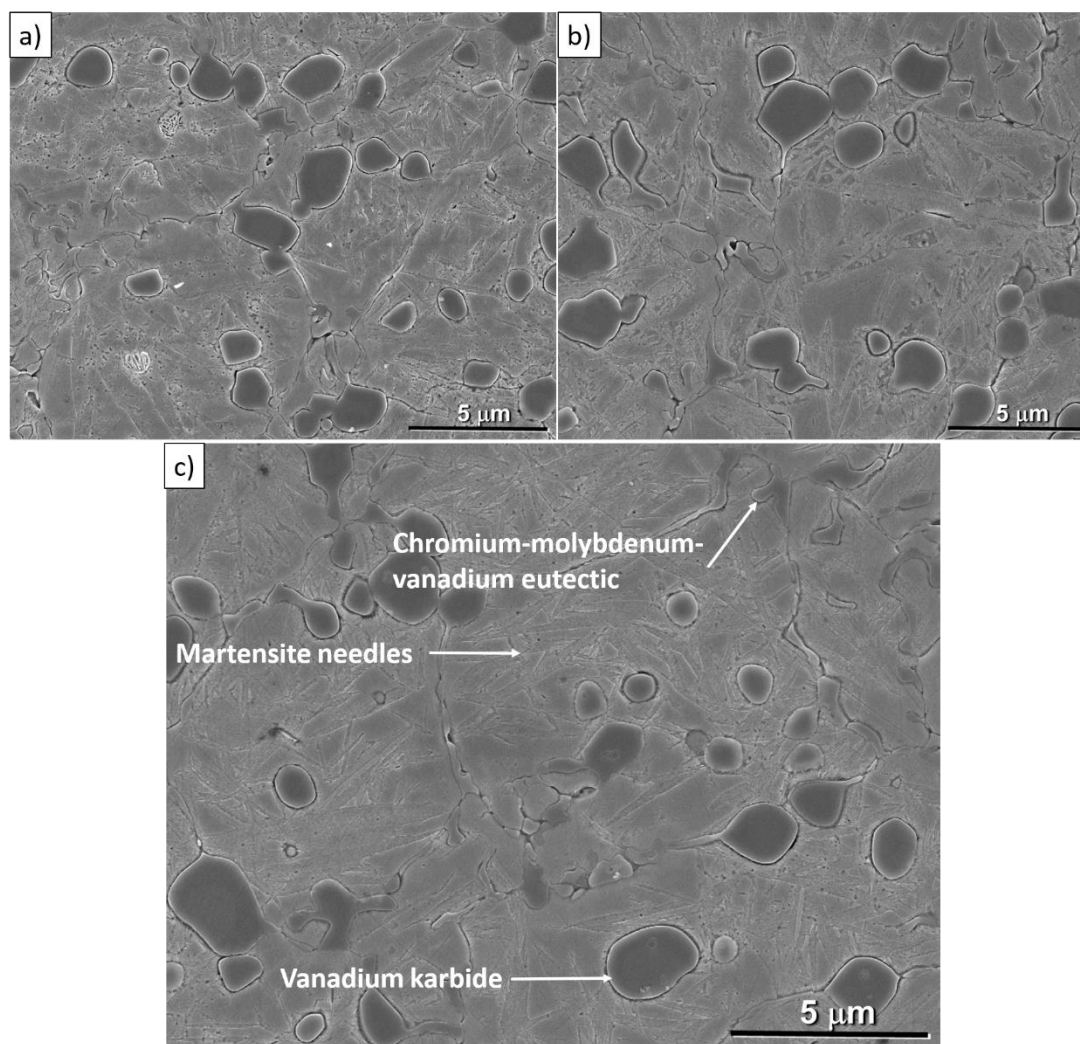


Figure 6. Microstructure of K390 steel upon mini-thixoforming; a) front part of the product; b) end of the product; c) middle part of the product.

Table 2. Fractions of microstructural constituents in K390 steel upon semi-solid processing, as determined by X-ray diffraction analysis. Measurements were performed on an automated powder diffractometer AXS Bruker D8 Discover with planar, positionally sensitive detector HI-STAR and a cobalt-ray lamp ($\lambda_{K\alpha} = 0.1790307$ nm).

K390	Phase	Content [wt. %]
	Austenite	31.3
	Fe_2VSi	16.9
	VC	15.5
	Martensite	10.8
	Fe_7V_3	9.1
	V_4WC_5	7.6
	Mo_2C	4.5
	Fe_7Co_3	4.3

5. Conclusions

The primary objective of this research was to produce an "inverted" microstructure which is profoundly different from all microstructure types known so far in semi-solid processed materials. When high-alloy tool steels are thixoformed, their final microstructure is typically strong but brittle, containing polygonal grains of super-saturated metastable austenite embedded in a carbide-austenite network. The ductile austenitic constituent deforms readily but, on the whole, the carbide-austenite network makes the material very brittle. For this reason, an alternative-type microstructure was sought which would eliminate or, at least, suppress these deficiencies. One of the solutions found involved the use of the high-alloy tool steel K390, which contains 8.94 % vanadium, for semi-solid processing. It is the high vanadium content which causes the highly-stable vanadium carbides to remain undissolved in the semi-solid state and enables them to greatly contribute to the alteration of the microstructure. To translate this concept into practice, a semi-solid processing technique was proposed which is now known as mini-thixoforming. Besides rapid heating, it also benefits from rapid solidification and cooling processes. Its parameters were experimentally optimized step by step. With the aid of calculations and experiments, a suitable heating temperature in the semi-solid range was found. At this temperature, the material can be thixoformed. Very small intricate-shaped products were obtained whose microstructures were substantially different from the microstructures obtained previously by semi-solid processing. These experimentally-produced microstructures consisted of a matrix of metastable and highly super-saturated austenite, in which globular vanadium carbides were embedded. The austenite also contained martensite and a small fraction of carbide-austenite eutectic. The best results were obtained using the route which involved heating to 1253 °C over 60 seconds and lateral extrusion into the shape die cavity for the product. Using these conditions, products with good surface quality and the desired "inverted" microstructures were obtained repeatedly.

Acknowledgments

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