

# CO-SPRAY FORMING AND TAILORED HEAT TREATMENT OF COMPOSITE TOOL STEELS

Chengsong Cui<sup>1</sup>, Alwin Schulz<sup>2</sup>, Dawid Nadolski<sup>3</sup>

<sup>1</sup> Foundation Institute for Materials Science, Badgasteiner Str. 3, 28359 Bremen, Germany

E-mail: [cscui@iwt.uni-bremen.de](mailto:cscui@iwt.uni-bremen.de), Website: [www.iwt-bremen.de](http://www.iwt-bremen.de)

<sup>2</sup> Foundation Institute for Materials Science, Badgasteiner Str. 3, 28359 Bremen, Germany

E-mail: [ashulz@iwt-bremen.de](mailto:ashulz@iwt-bremen.de), Website: [www.iwt-bremen.de](http://www.iwt-bremen.de)

<sup>3</sup> Foundation Institute for Materials Science, Badgasteiner Str. 3, 28359 Bremen, Germany

E-mail: [nadolski@iwt-bremen.de](mailto:nadolski@iwt-bremen.de), Website: [www.iwt-bremen.de](http://www.iwt-bremen.de)

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## ABSTRACT

During micro cold forming the tools are loaded differently in various functional areas, tailored material properties in the specific regions are therefore required. To meet this requirement, different tool steels can be applied in the specific regions of the tools, with a good bonding in between. Such composite materials can be produced by a newly developed spray forming process. Moreover, the different tool steels in the composite tools may require different heat treatment conditions to achieve optimal material properties. For this purpose, a tailored heat treatment based on middle frequency induction heating has been developed to austenitize the tool steels at different temperatures. The research results showed that fine and homogeneous microstructures could be achieved in both tool steels, with a gradual transition of chemical and physical properties in between. The microstructures and properties of the composite material are clearly correlated with their austenitization conditions during the induction heating.

## 1 INTRODUCTION

During micro cold forming the tools are loaded differently in various functional areas, tailored material properties in the specific regions are therefore required [1-4]. To meet this requirement, different materials can be applied in the specific regions of the tools. To reduce critical stresses at the interface during heat treatment, an excellent bonding of the materials can be achieved by the use of a gradual material transition in between. In addition, the tool materials need to be micro-machinable since the geometrical structures of the tool surfaces are smaller than 1 mm in more than one dimension. Large carbides or significant segregation in the tool materials should be avoided since they would disturb precision machining of the tools.

This kind of composite tool material has been produced by a new spray forming process [5]. During this process, two different tool steels are melted in two crucibles separately. One steel melt is poured first into a tundish, followed by gas atomization and spray deposition on a rotating tubular substrate, resulting in a ring-shaped deposit (inner ring). When the first crucible is empty, the second steel melt is poured into the tundish, gas-atomized and spray-deposited over the inner ring to form an outer ring. If there is still residual of the first melt in the tundish when the second melt is added, they mix in the tundish and the proportion of the second steel increases gradually as it is continuously added. Consequently, a gradual transition of chemical composition is generated inside the composite deposit. In addition, fine and homogeneous microstructures can be achieved in the composite deposit due to rapid solidification associated with spray forming [6, 7].

Moreover, the different materials in the composite product require different heat treatment conditions to achieve optimal material properties. For example, the recommended austenitization temperatures for high-speed steels are much higher than those for cold work tool steels [8, 9]. The traditional heat treatment in furnace (at uniform austenitization temperatures) is not suitable for the composite materials. To overcome this problem, a tailored heat treatment is to be developed. One

approach to achieving tailored austenitization is to use middle frequency induction heating: the composite material is heated to a lower austenitization temperature (e.g., for cold work tool steels) and hold for a period by oscillating the inductor over the complete sample, followed by the oscillation of the inductor only over the region where a higher austenitization temperature (e.g., for high-speed steels) is required. The heating rate of the composite material can be adjusted by the induction power input and the oscillation speed of the inductor. In this way, a tailored austenitization of the composite material can be realized.

In this study, a ring-shaped composite deposit (inner: high-speed steel HS6-5-3C (AISI M3:2); outer: hypereutectoid cold work tool steel (X110CrMoV8-2) with higher chromium content) was produced by the newly developed spray forming process. The deposit was further hot rolled to eliminate porosity and break eutectic carbides. Finally, the composite material was heat treated by means of induction heating to achieve optimal microstructures and material properties. The microstructures and mechanical properties of the composite tool material have been investigated and evaluated.

## 2 EXPERIMENTALS

### 2.1 Material

A high-speed steel HS6-5-3C (AISI M3:2) and a cold work tool steel X110CrMoV8-2 were selected in this study for the manufacture of composite material. The nominal chemical composition of the two steels is listed in Table 1 [8, 9]. A large number of V-rich carbides in HS6-5-3C lead to high hardness and excellent wear resistance. X110CrMoV8-2 has lower content of hard carbides and significantly higher toughness than HS6-5-3C, and it is thus much easier to be machined. These two steels are supposed to be applied in different functional zones of micro infeed rotary swaging tools, i.e., reduction zone and calibration zone [5]. A gradual material transition is to be generated between the two zones by means of the newly developed spray forming process.

Steel <sup>*)</sup>	C	Cr	W	Mo	V
HS6-5-3C	1.3	4.2	6.3	5.0	3.0
X110CrMoV8-2	1.0	8.0	–	2.5	0.3

<sup>\*)</sup> short names according to EN 10027-1

Table 1: Nominal chemical composition of high-speed steels HS6-5-3C and X110CrMoV8-2.

### 2.2 Spray forming

The spray forming of the composite material was carried out on the Sk1+ facility at the University of Bremen, Germany (see Fig.1). The steels were melted separately in two crucibles. HS6-5-3C was firstly poured into a tundish to a certain fluid level and atomized by nitrogen gas jets generated by means of a free-fall type gas atomizer. A tubular substrate (114 mm OD×103 mm ID ×812 mm) of carbon steel was used to collect the atomized droplets. It rotated at a speed of 1.52 revolutions per second. The initial spray distance from the gas atomizer to the substrate was 490 mm. The atomized droplets spray-deposited on the rotating substrate and formed a ring-shaped deposit (inner ring). Subsequently, X110CrMoV8-2 melt was poured into the tundish and spray-deposited over the inner ring to form an outer ring. Since the two melts mixed partially in the tundish, a gradient zone was generated between the two rings. The pouring temperatures of HS6-5-3C and X110CrMoV8-2 were 1592 °C and 1568 °C, respectively. The diameter of the pouring nozzle assembled in the base of the tundish was 6.5 mm, corresponding to a mass flow rate of approximately 0.47 kg/s. The atomization gas pressure was 0.35 MPa for both melts, corresponding to a mass flow rate of approximately 0.25 kg/s each. Thus the gas to melt ratio GMR was about 0.53 for both melts. To achieve a relatively flat contour of the deposit, the gas atomizer scanned in the axial direction of the substrate (see Fig. 1a). The scanning angle of the atomizer was 8°, and the scanning frequency was 5 Hz.

The spray-formed deposit was in the form of a ring-shaped product, with an outer diameter of about 335 mm and an inner diameter of 114 mm (see Fig. 2a). To reduce hardness and remove stresses, the spray-formed deposit was soft annealed: heating to 840 °C at the rate of 50 K/h, holding for 4 h, followed by cooling to 500 °C at the rate of 20 K/h and uncontrolled cooling in the turned-off furnace to room temperature.

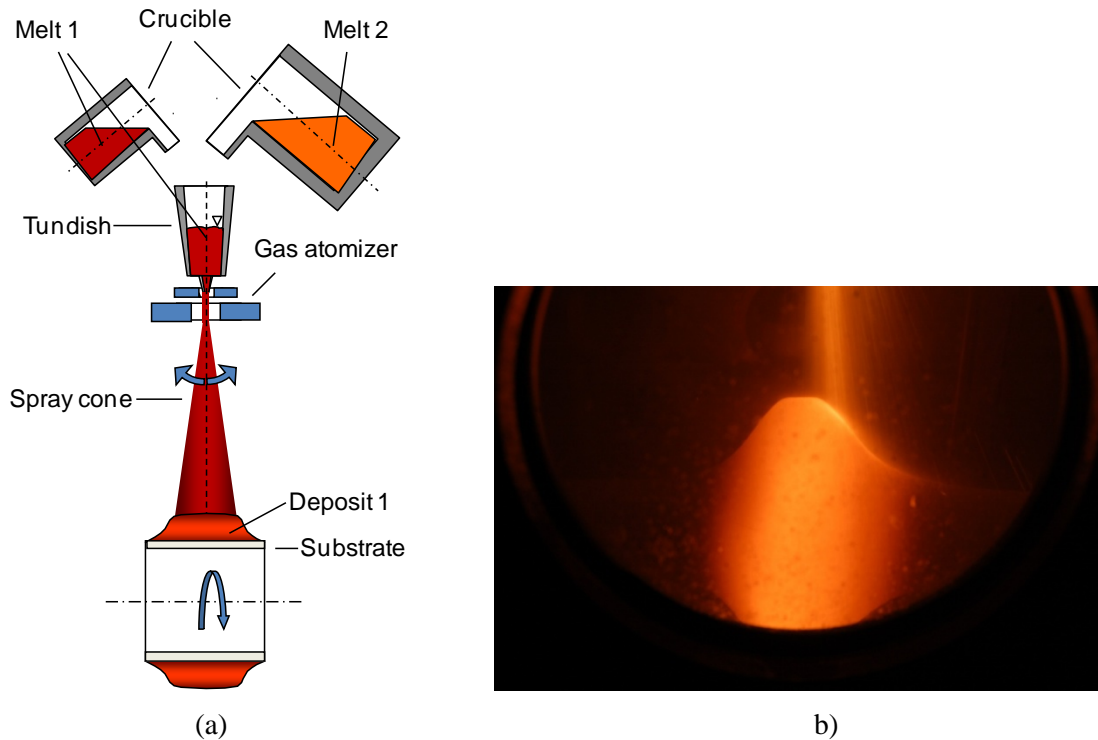


Figure 1: a) schematic of co-spray forming of a ring-shaped composite deposit and (b) photo of the spray forming process.

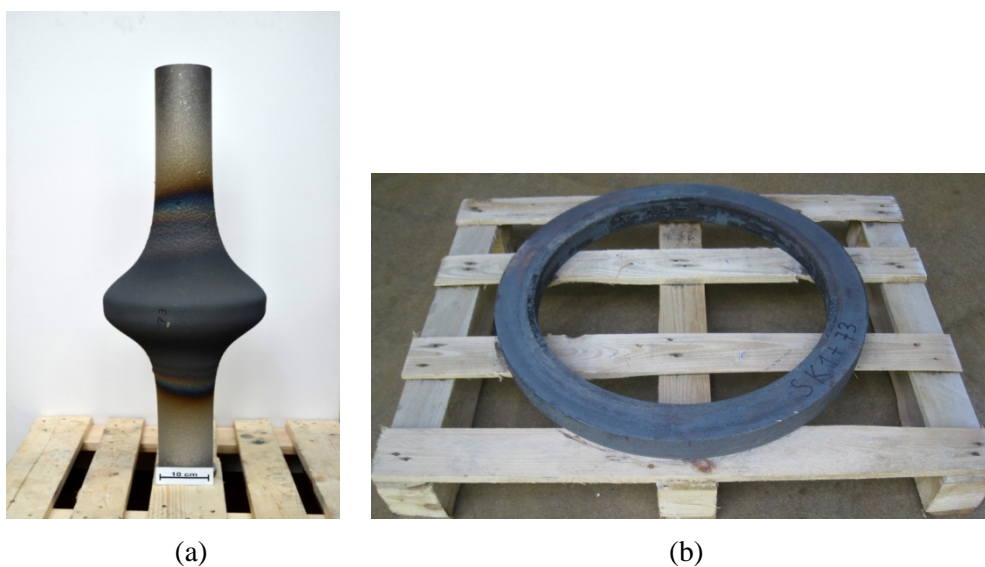


Figure 2: a) the ring-shaped graded deposit and b) the hot rolled ring.

## 2.3 Hot rolling

The spray-formed deposit was subsequently excised into pieces. The middle part of the deposit was machined to a ring preform (318 mm OD×125 mm ID×50 mm), and forwarded to a rolling mill for ring rolling. The sample was preheated in a furnace to 1100 °C and rolled to the final dimension (543 mm OD×431 mm ID×39 mm). The thickness of the hot rolled ring was about 55 mm. The thickness reduction ratio of the ring was 1.72 and the area reduction 2.23. The length change of the ring was 1.71 at the outer circumference and 3.44 at the inner circumference. After ring rolling the sample was cooled slowly in furnace from 860 °C to room temperature. A picture of the hot-rolled ring is shown in Fig. 2b. Cylindrical samples (∅20 mm×50 mm) were machined along the thickness of the ring for the tailored heat treatment.

## 2.4 Induction heat treatment

The tailored heat treatment of a composite material is illustrated in Fig. 3. An oscillating ring-shaped inductor is used to heat the cylindrical samples. As shown in Fig. 3a, the upper region of the sample is Alloy 1 and the lower region is Alloy 2. Between the two alloys is the gradient zone. The desired austenitization temperatures for Alloy 1 and Alloy 2 are T1 and T2, respectively. Middle frequency induction was applied for a thorough heating from the surface to the core of the sample. The induction heating process is divided into three stages: 1) the complete sample is firstly heated up to the temperature T2 by oscillating the inductor over the complete sample (Phase 1); 2) the inductor continues to oscillate over the complete sample but the inductor current is reduced, so that the sample temperature is hold at T2 (Phase 2); 3) the inductor oscillates only over the upper region (Alloy 1) and the local temperature is further increased to T1 (Phase 3). In order to hold the temperature of the lower region at T2 in Phase 3, the inductor should also oscillate over the complete sample after several times of oscillation over the upper region. This combined oscillation is repeated and a tailored induction heating can be achieved (see Fig. 3b and 3c).

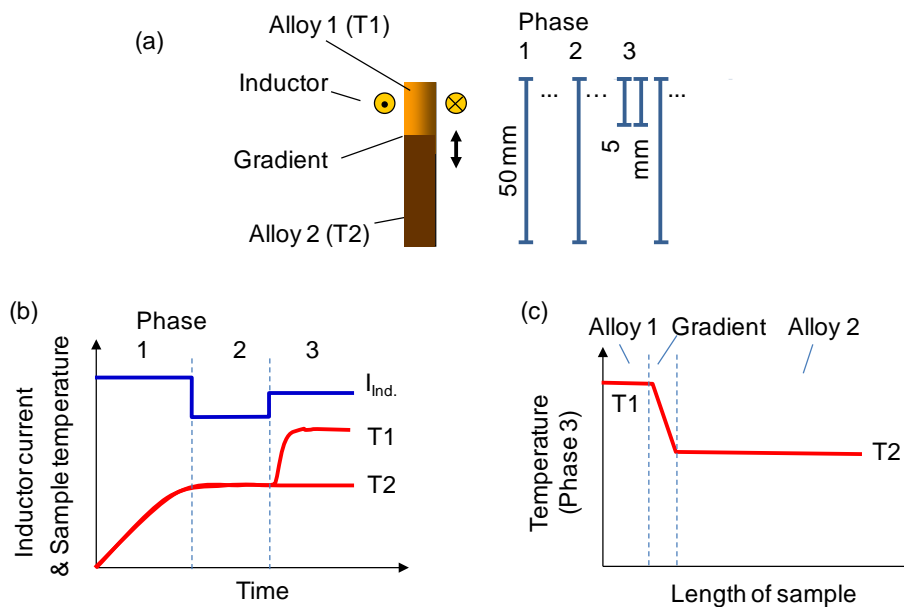


Figure 3: Tailored austenitization of a composite material based on middle frequency induction heating and oscillation of the inductor.

Figure 4 shows the setup of the induction heat treatment of the composite material. The induction heat treatment facility is a VL1000 SINAC 200/300 S MFC with Multi-Frequency-Concept from EFD. In this study, only the middle frequency induction was used. The inductor was a water-cooled single turn copper inductor with an inner diameter of 25 mm, a cross section of 6×6 mm<sup>2</sup>, and a wall

thickness of 1 mm. To reduce heat loss, thermal insulation (glass fiber) was applied at both ends of the sample. Sheath thermocouples ( $\varnothing 1$  mm, type K) were used to measure the inner temperature at various positions of the sample. Six  $\varnothing 1.1$  mm holes of different depths were bored at half radius of the sample for placing the thermocouples. A traditional pyrometer (KTR 1075 from Maurer) and a 2-color (ratio) pyrometer in combination with a video camera with a short wavelength infrared filter (ISR 6-TI Advanced, LumaSense) were used to measure the surface temperature of the sample during the heat treatment. In addition, a video camera was used to record the induction heat treatment process.

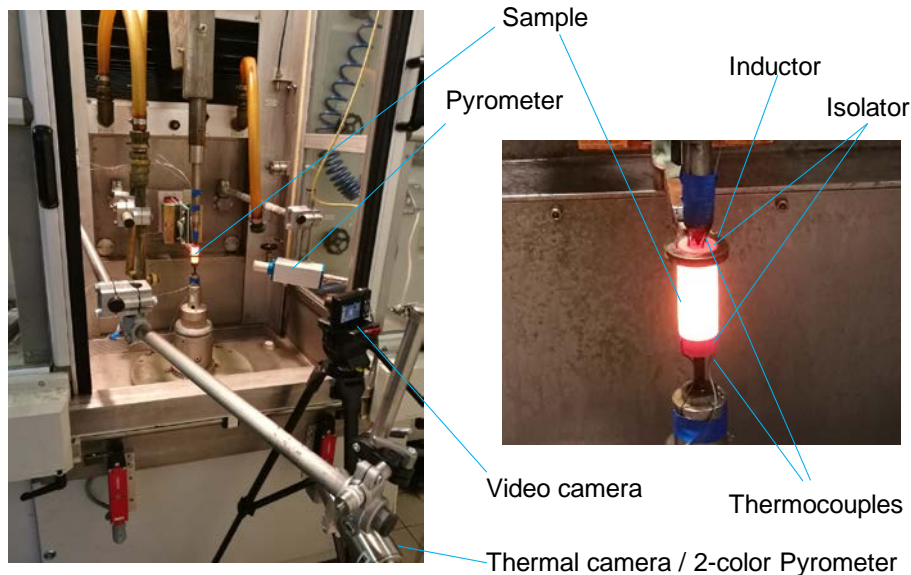


Figure 4: Set up of induction heat treatment of the composite material.

The desired austenitization temperature for HS6-5-3C and X110CrMoV8-2 is about 1180 °C and 1080 °C, respectively. To achieve the desired temperatures, the induction power input and the oscillation of the inductor were adjusted and optimal process parameters were determined, as listed in Table 2. The induction frequency used for the process is about 14 kHz. The moving speed of the inductor was kept constant at 4000 mm/min. In phase 1 the inductor oscillated over the complete sample for 33 times, and the inductor current was 2.0 kA. In phase 2 the inductor continued to oscillate over the complete sample for 60 times, and the inductor current was reduced to 1.4 kA. In Phase 3 the inductor oscillated two times over the upper region (5 mm) of the sample, followed by one oscillation over the complete sample. This combined oscillation was repeated 30 times, and the inductor current was increased to 1.6 kA. The power input in the inductor for the three phases was 11.0 kW, 3.6 kW and 5.0 kW, respectively.

Process parameter	Phase 1 (heating)	Phase 2 (holding)	Phase 3 (further heating)
Number of oscillation	33	60	(2+1)×30
Region of oscillation	complete sample (50 mm)	complete sample (50 mm)	upper region (5 mm) + complete sample (50 mm)
Moving speed of inductor (mm/min)	4000	4000	4000
Inductor current (kA)	2.0	1.4	1.6
Power (kW)	11.0	3.6	5.0

Table 2: Process parameters for a representative induction heating of the composite material.

Thermal history and temperature distribution of a composite sample processed under the abovementioned condition are presented in Fig. 5. Six pairs of thermocouples were embedded at various positions of the sample, as illustrated in Fig. 5a. For example, the thermocouple T1 measured the temperature at the position 5 mm from the top of the sample, while the thermocouple T6 measured the temperature at the position 40 mm from the top of the sample. It is clearly seen that the sample was heated homogeneously to approximately 1070 °C in the first phase and the temperature difference among the various positions was small. In the second phase the temperature in the middle region (T2, T3, and T4) was relatively constant, but the temperature at the both ends of the sample was lower due to more heat loss. The temperature difference between the end and the middle section of the sample was about 50 °C. To further reduce the temperature inhomogeneity one should improve the thermal insulation at the ends of the sample and/or compensate the heat loss by the use of additional heating. In the third phase the temperature at the upper region of the sample (T1 and T2) further increased to 1170-1180 °C, while the temperature at the lower region (T5 and T6) was kept relatively constant. In between a temperature gradient was generated. The temperature distribution of a composite sample in Phase 3 is also clearly seen in Fig. 5b (thermal image generated by ISR 6-TI). Afterwards, the induction power input was shut down, and the sample was cooled in air. Since the tool steels can be hardened in air, traditional quenching medium was not applied in this study. Finally, the composite sample was tempered under normal conditions: three times in a vacuum furnace in a protective gas atmosphere at 550 °C for 2 hours.

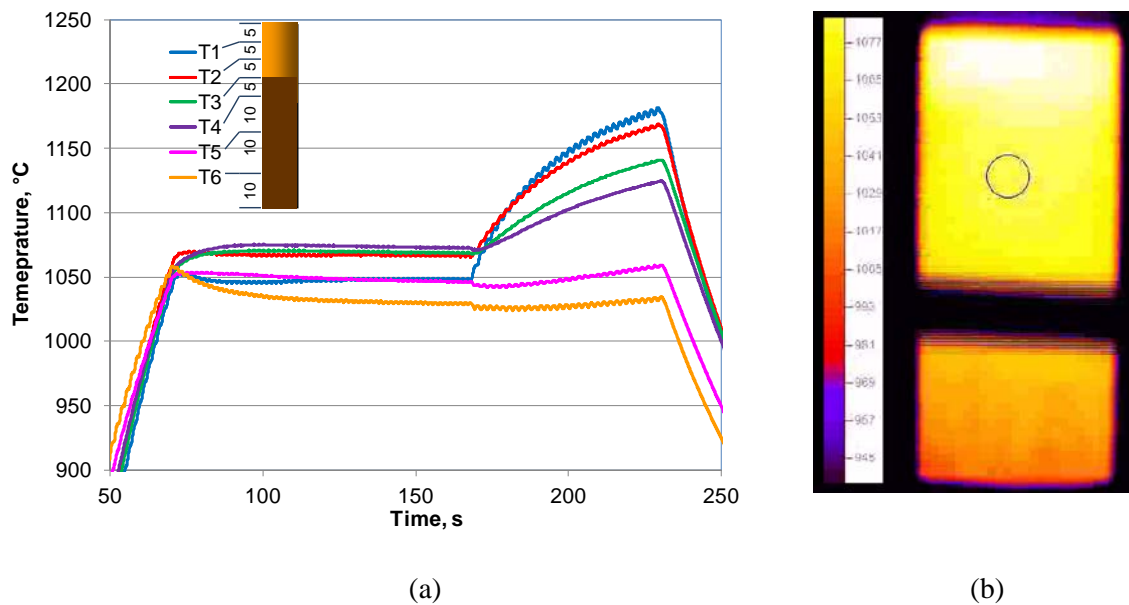


Figure 5: a) Thermal history and b) temperature distribution of a composite sample processed by mean of induction heating (dark bar: inductor, circle: measurement area of the 2-color pyrometer, color bar: temperature in °C).

## 2.5 Analysis and testing of materials

Microscopic samples were taken from the spray-formed deposit, the hot rolled material, and the induction hardened sample. They were investigated by means of optical microscopy (Axiophot, ZEISS). The etchant Nital (3% alcoholic HNO<sub>3</sub> at RT) was used to reveal the microstructures of the micrograph samples. Macrostructures of the samples were revealed by the mixture of HCl, HNO<sub>3</sub> and H<sub>2</sub>O (700 ml HCl : 300 ml : HNO<sub>3</sub> : 1000 ml H<sub>2</sub>O). Hardness of the samples was examined by Rockwell indentation measurement.

Chemical composition of the hot rolled ring was analyzed by spark optical emission spectroscopy (S-OES, ARL3460). The sample was ground at intervals, starting from the inner surface of the ring,



and the S-OES measurement was conducted at the newly ground surfaces. In this way, the distributions of the chemical elements in the thickness of the composite material were determined.

### 3 RESULTS AND DISCUSSION

#### 3.1 Material characterization of the composite deposit

Figure 6 presents a macro-etched transverse section of the composite deposit as well as the micrographs of porosity in the deposit. The inner ring (HS6-5-3C) of the deposit appears darker than the outer ring (X110CrMoV8-2) due to stronger chemical etching of the steel with higher content of carbides. The interface between the two steels is located at the position about 30 mm from the inner surface of the deposit.

Tree-ring structures have been observed in the deposit after macro-etching. This indicates that the deposit was spray-formed under a relatively cold condition and the deposit surface might be more or less completely solidified and did not experience a sufficient re-melting process as new layers were deposited on. As a result, the interfaces between the layers are porous and they are revealed as dark rings by chemical etching. The tree-rings become more pronounced in the outer area of the deposit since the deposit surface for heat exchange are larger, the deposit layers are thinner, and they are more intensively cooled.

In higher magnification the pores of the deposit show irregular shape. They are generally called interstitial porosity or cold porosity [4, 5]. This kind of porosity is formed under relatively cold spray forming condition when the interstices between the droplet fragments and solid particles in the surface area cannot be refilled by sufficient liquid phase and local solidification shrinkage occurs. Larger pores are found mostly in the outer area of the deposit since the cooling rate of the deposit in the outer area is faster than in the inner area, as mentioned above.

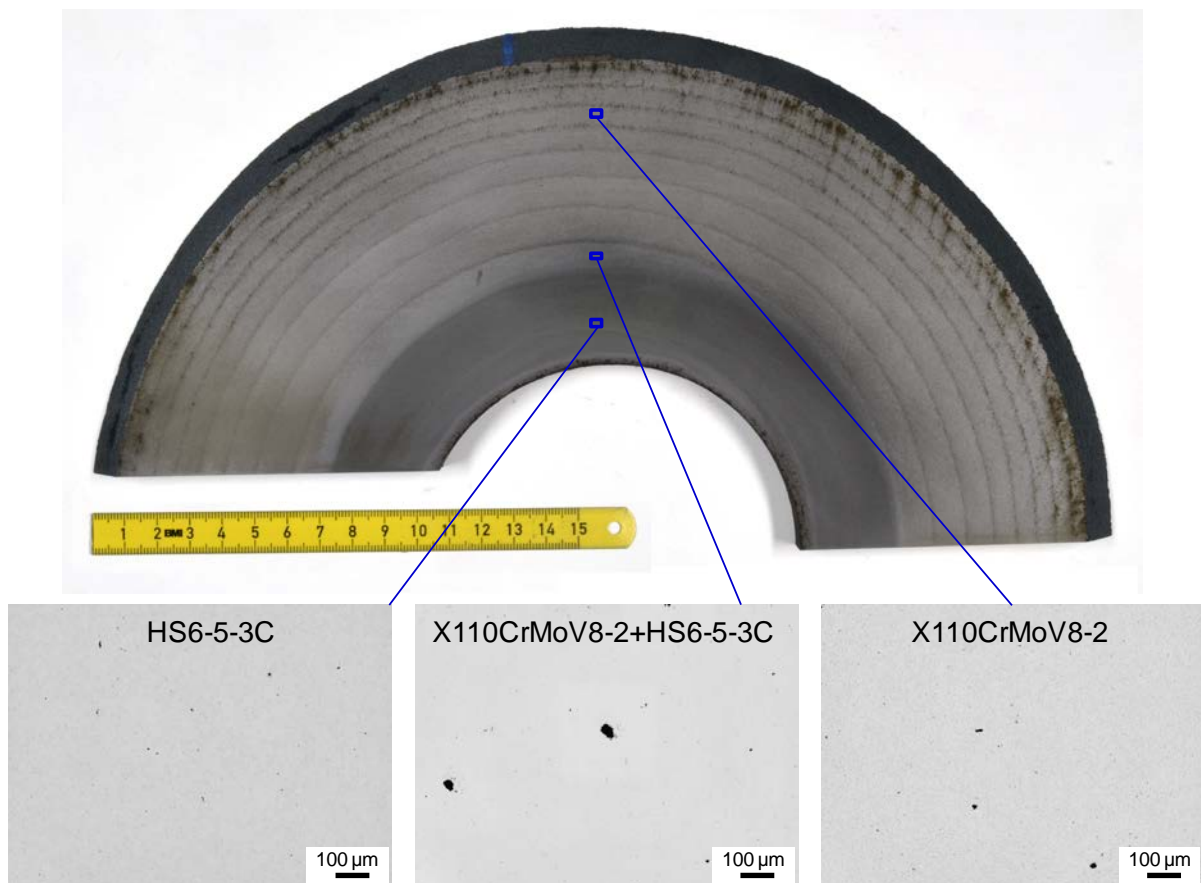


Figure 6: Transverse section and porosity of the composite deposit with an inner ring of HS6-5-3C, an outer ring of X110CrMoV8-2, and a gradient zone in between.

### 3.2 Element distributions in the hot rolled material

Figure 7 shows the distributions of the main alloying elements C, Cr, Mo, W and V in the hot rolled ring. The inner area is the high-speed steel HS6-5-3C and the outer area is the cold work tool steel X110CrMoV8-2. The carbon content is approximately 1.3 wt.% in the HS6-5-3C and 1.0 wt.% in the X110CrMoV8-2. There are much more hard carbide forming elements Mo, W, and V in the HS6-5-3C region than in the X110CrMoV8-2 region. The content of eutectic carbide forming element Cr is significantly higher in the latter. The gradient zone starts at the position about 12 mm from the inner surface of the ring. It is clear that the slope of the gradient of the elements depends on the level of mixture of the two steel melts in the tundish. If there is less melt of the first steel in the tundish when the melt of the second steel is added, the slope of the gradient would be steeper.

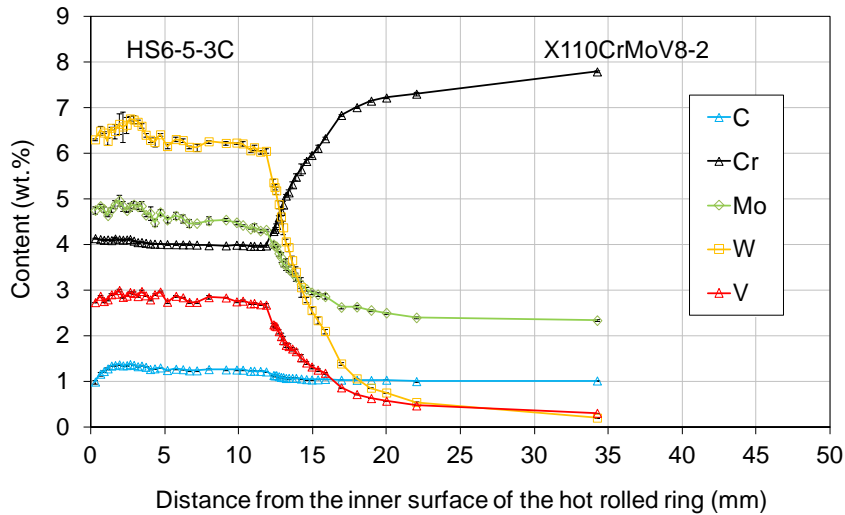


Figure 7: Distributions of main chemical elements in the hot rolled ring measured by spark optical emission spectroscopy (S-OES).

### 3.3 Material characterization of the as-rolled ring

Figure 8 presents the macrostructure and the microstructures of the as-rolled ring. The tree-ring structures are still observed in the macro-etched sample. However, the porosity in the composite deposit has been significantly reduced by hot rolling. There is a small amount of porosity in the transition zone since the deformation ratio of the spray-formed ring is not sufficiently high. The tree-ring patterns should be relevant to the different grain structures in the sample, as a consequence of deformation and dynamic recrystallization of different degrees, corresponding to different porosity level in the as-spray-formed ring. The microstructures are composed of ferrite and carbides (as-annealed structures due to slow cooling of the hot rolled material in furnace). The carbides are homogeneously distributed in the hot rolled material. The eutectic carbide frequently observed at the grain boundaries of as-spray-formed deposits has been broken up to some extent due to hot rolling. The V-rich carbides are observed in the inner area (HS6-5-3C) and more Cr-rich eutectic carbides in the outer area (X110CrMoV8-2). The size of the blocky MC carbides is about several microns. A gradual transition of the microstructure has been observed between the inner area and the outer area.



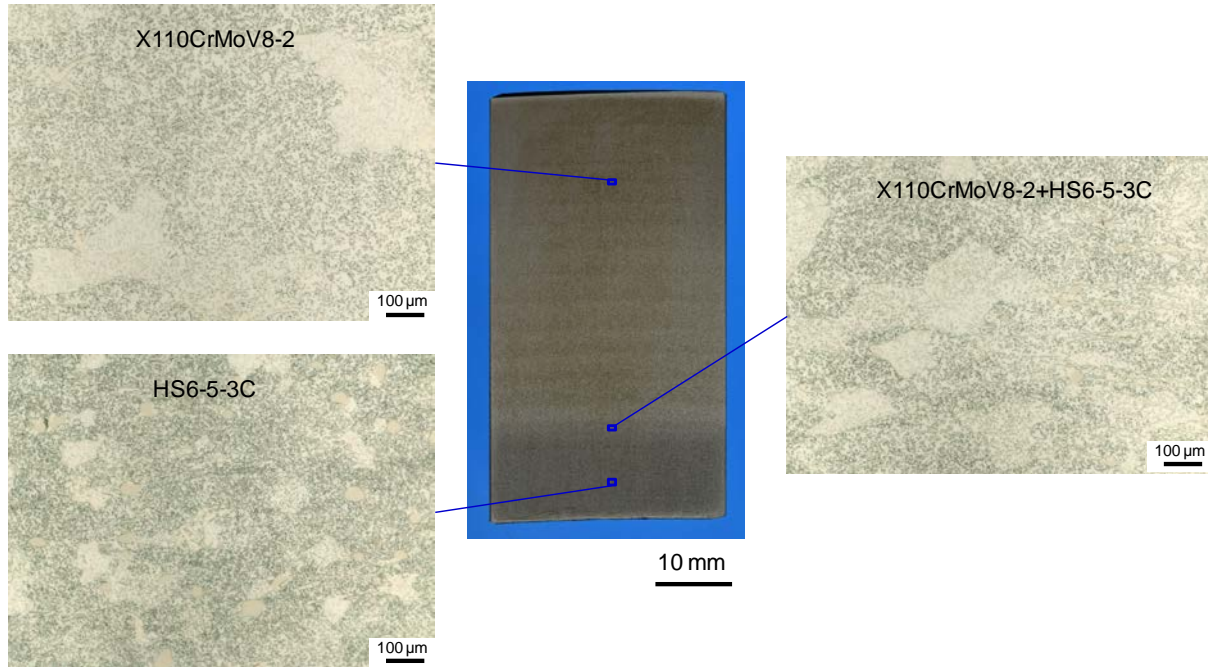


Figure 8: Macrostructure and microstructures of the as-rolled composite material.

### 3.4 Microstructures of the composite material after induction heat treatment

The microstructures of the hot rolled composite material after hardening and tempering are presented in Fig. 9. The primary austenite grain structures are clearly revealed in the as-hardened sample. These fine and homogeneous grain structures are typically found in spray-formed materials. The MC type carbides look grey. After tempering the composite material, the micrographs look darker because of stronger etching effect of the fine carbides in the matrix. The primary carbides and the eutectic carbides appear white under this condition. The eutectic carbides in the X110CrMoV8-2 region are also clearly revealed.

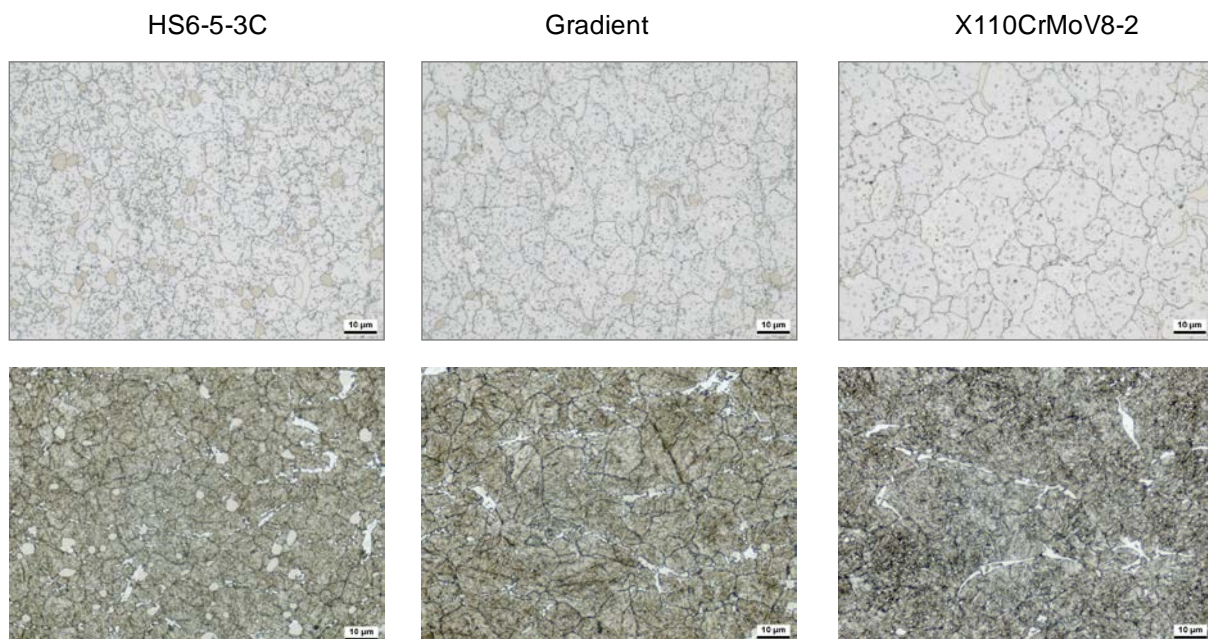


Figure 9: Microstructures of the composite material (upper: after induction hardening; lower: after induction hardening and tempering)

### 3.5 Hardness of the composite material after induction heat treatment

The hardness profiles of the composite material after induction heat treatment are presented in Fig. 10. The maximum austenitization temperatures reached in the various regions of the composite material are also plotted in this diagram. It shows that the desired gradient temperature is achieved. The maximum austenitization temperature is about 1180 °C in the HS6-5-3C region and 1060 °C in the X110CrMoV8-2 region. After induction hardening the hardness of the HS6-5-3C steel and the X110CrMoV8-2 steel can reach 65 HRC. However, the hardness of the transition zone is low (for example, about 52 HRC at the length position of 17 mm). It is known that the recommended austenitization temperature for X110CrMoV8-2 is 1080 °C [9]. If the actual austenitization temperature is higher (as seen in the transition zone in Fig. 10), more carbides dissolve in the matrix and the martensite transformation temperature is lowered, leading to higher content of retained austenite and thereby lower hardness. Fortunately, the hardness in the transition zone was increased after the tempering for three times at 550 °C, due to precipitation of fine carbides and further transformation of retained austenite to martensite. It is seen in the figure that the hardness is in the range of 62-65 HRC in the HS6-5-3C region and in the transition zone, while the hardness of the X110CrMoV8-2 region is in the range of 56-62 HRC. The hardness of the composite materials is clearly correlated with their austenitization conditions during the induction heating.

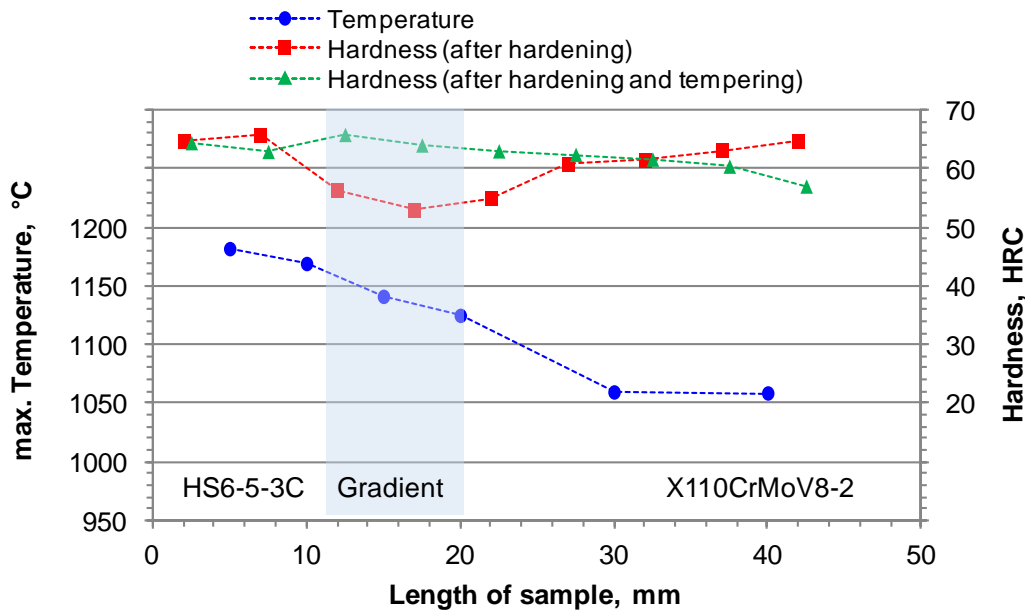


Figure 10: Maximum austenitization temperature and hardness of the composite material.

## 4 CONCLUSIONS

1) Composite tool steels have been produced by means of a newly developed spray forming process. During this process two different tool steels (HS6-5-3C and X110CrMoV8-2) were melted separately and spray-deposited on a rotating tubular substrate one after another, resulting in a ring-shaped composite deposit when the two melts partially mixed in the tundish. Consequently, different microstructures and mechanical properties were combined in a single deposit with a gradual transition in between.

2) A tailored heat treatment for the composite material has been developed based on middle frequency induction heating and oscillation of the inductor: the composite material was heated to a low austenitization temperature (e. g., desired for cold work tool steels) and hold for a period by oscillating the inductor over the complete sample, followed by the oscillation of the inductor only over the region where a high austenitization temperature (e.g., desired for high-speed steels) was required. The heating

rate of the composite material could be adjusted by the induction power input and the oscillation speed of the inductor. In this way, a tailored austenitization of the composite material was realized.

3) The results showed that fine and homogeneous microstructures could be achieved in both regions of the composite material, with a good bonding by the gradient zone in between. The hardness of the HS6-5-3C steel and the X110CrMoV8-2 steel can reach the desired values 65 HRC and 60 HRC, respectively. The microstructures and properties of the composite materials are clearly correlated with their austenitization conditions during the induction heating.

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