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Surface Modification and Wear Properties of Direct Metal Laser Sintered Hybrid Tools Used in Moulds

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Injection moulding is one of the most productive plastic forming processes. Product development and the reduction of production time require new solutions in tooling design and manufacturing. Direct metal laser sintering (DMLS) allows moulds to be built with special cooling systems, which offer curved cooling lines that can follow the geometry of the part (conformal cooling). One disadvantage of DMLS, its high cost, can be dramatically reduced with the building of hybrid structures. With conventional tool steels as the base plate and only the special geometry of the part sintered on the top, the final geometry can be manufactured after sintering by conventional process technologies. We produced hybrid structures by direct metal laser sintering maraging steel (MS1) powder onto the surface of commercial mould steels and studied the effect of different heat treatments on porosity, tribological behaviour and the microstructure. The transition zone was also characterized.

Keywords: direct metal laser sintering, hybrid structure, maraging steel, 1.2343 steel, heat treating, wear

Highlights

- *Age hardening and quenching was compared on the hardness of the different steel grades. Age-hardening results in almost the same hardness as quenching in maraging steels, while the same heat treatment resulted in softening in the case of 1.2343 steel.*
- *Quenching produced the same hardness as age hardening in MS1 steel.*
- *The sintered maraging steel showed lower wear resistance than its conventionally produced counterpart in all cases, which can be explained with the higher porosity as a result of sintering.*
- *In the case of hybrid structures, it is necessary to select heat treatments and heat-treating parameters that produce the required properties in both components.*
- *If tool steel is used in hybrid constructions, which require quenching to assure the prescribed properties, this heat treatment can produce the same hardness in the MS1 part as age hardening can when high-temperature tempering is applied after quenching.*

0 INTRODUCTION

Injection moulding is one of the most critical polymer processing technologies. The most significant phase of the injection moulding cycle is cooling time, which can be more than half the whole cycle [1]. One of the best ways to achieve a reduction in cooling time is to use mould inserts with conformal cooling [2]; shrinkage and warpage can thus also be decreased [3].

With layer manufacturing technologies, parts of complex internal structures can be built, which can be a major advantage for injection moulds [4]. The cooling channels may have a complex form, which cannot be manufactured with conventional methods, such as drilling or milling [5]. Tooling companies are increasingly employing additive manufacturing (AM) technologies to fabricate moulds with integrated conformal cooling channels. Selective laser sintering (SLS) is an AM technique, which is capable of manufacturing 3D parts from different powder materials [6]. The powder is scanned and selectively sintered according to the two-dimensional cross-section of the sliced model of the CAD geometry. In the beginning, these systems only worked with polymer powders. Then, as the technology developed, companies introduced laser sintering processes for building parts from metal powders. Direct metal laser sintering (DMLS) [7] and selective laser melting (SLM) [8] are the most widespread techniques at present, and both technologies use metal powders without coating [9].

Producing mould inserts from metal powders by AM is an expensive technology in comparison to conventional machining. A major challenge is to integrate free shape AM and cheap conventional manufacturing processes [10]. Metal AM is a promising technology to fabricate complex geometry and hybrid structures from different metals, e.g. cobalt-based alloy deposited on conventional tool steel [11] or sintering of maraging steel on a copper substrate [12].

A limited number of tool steels are available for additive manufacturing; maraging steel (1.2709) powder is the regularly used raw material for

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printing moulds. Maraging steels are strengthened by precipitation hardening in furnaces without a protective atmosphere and at relatively low temperatures (approx. 500 °C). Surface heat treatment, such as nitriding, can reduce wear and corrosion, and the lifetime of plastic injection moulds can be extended. Nitriding is a surface-hardening process, introducing nitrogen into the surface of the steel. In conventional gas nitriding processes, the temperature range of nitriding is 500 °C to 550 °C, which is higher than the aging temperature of maraging steels, and thus would result in over-aging. Different thermomechanical heat treatments such as nitriding [13], nitrocarburizing, boriding and carburizing can improve the wear resistance of maraging steel [14]; the best results can be achieved by plasma nitriding [15].

In the case of hybrid tooling, the base part is manufactured with conventional technologies, while the upper part (designed for conformal cooling) is built layer by layer with DMLS technology. In an optimal case, the base and the over-sintered material have the same chemical composition. Maraging steels have a relatively high price and only a limited range of raw materials is available; therefore, they are regularly replaced by conventional tool steels (1.2311, 1.2343, etc.) and heat treated similarly to the base part [16].

Samples made from DMLS MS1, Böhler W722 and 1.2343 steels with different surface hardness after ageing, hardening, nitrocarburizing and nitrocarburizing combined with post-oxidation (oxy-nitriding) were investigated. The microstructure, hardness and wear properties of the samples were characterized; the hybrid structures were studied and analysed mainly from a tribological point of view but also with a focus on the transition layer.

1 EXPERIMENTAL

1.1 Materials

The samples were prepared from “MaragingSteel MS1” steel powder by direct metal laser sintering (DMLS). Injection moulds having conformal cooling channels are regularly made from MS1. EOSINT M270 (200 W) was used with the EOS MS1_Surface 1.0 parameter setting and a layer thickness of 20 µm. Commercially available materials were selected as reference: Bohler-Uddeholm W722 VMR (~1.2709) maraging steel made by vacuum induction melting, and 1.2343 steel, a material generally used for injection moulds (Table 1).

1.2 Sample Manufacturing

Disk-like specimens with a diameter of 30 mm and a thickness of 5.5 mm were produced. Samples made of MS1 powder were sintered, while the reference materials were machined from a rod (Ø30 mm). To guarantee the same test conditions, the specimens were ground then polished with the same parameters with 1 µm diamond paste (surface roughness, Ra = 0.015 µm to 0.02 µm) before the heat treatments. The samples were repolished after age-hardening, but they were not repolished after nitro-carburizing and oxy-nitriding. Hybrid test specimens were also made by sintering MS1 material onto the surface of rectangular W722 and 1.2343 blocks.

1.3 Heat Treatment

Sintered parts (MS1) have a surface hardness of 33 HRC to 37 HRC [19], but injection moulds require a much harder material. Surface hardness can be increased by heat treatments, especially by surface hardening methods, like carbonitriding. MS1 and W722 grades can be hardened by precipitation treatments. Heat treatment parameters, generally applied during precipitation or age hardening are as follows: the heating temperature range is 455 °C to 510 °C, while the holding time is 3 to 12 hours. Then the metal is slowly cooled to room temperature in air [20]. The results of several heat treatments showed that the above-mentioned temperature-time parameters only produced a small, 1 HRC to 2 HRC increase in hardness [21]. In the case of MS1 and W722 steels, nitriding and age hardening are carried out in one step.

Bulk and surface heat treatments were used to achieve higher strength and hardness of the steels and enhance the wear resistance of their surface. The technological parameters of age hardening and

Table 1. *Chemical composition of the tested steel materials [17] to [19]*

Steel	C	Cr	Ni	Mn	Si	Al	Co	Mo	Ti	V
MS1	<0.03	<0.5	17 to19	<0.1	<0.1	0.05 to 0.15	8.5 to 9.5	4.5 to 5.2	0.6 to 0.8	
W722	<0.005		18				9.25	4.85	1.00	
1.2343	0.38	5.00		0.4	1.10			1.30		0.4

Table 2. *Heat treatment conditions*

Heat treatment method	Abbrev.	Material	Temperature [°C]	Duration [h]	Atmosphere
Age hardening	AH	MS1, W722 (1.2343)	500	4	air
Quenching + 2 × tempering	Q	MS1, 1.2343	1050	1	vacuum
			530 and 480	530 and 480	
Nitro-carburizing	NC	MS1, W722, 1.2343(Q)	550	8 (∑12)	mixture of gases
Oxy-nitriding	NO	MS1, W722, 1.2343(Q)	550	7 (∑13)	mixture of gases 6 l/h to 7 l/h water
			450	1	

quenching were selected based on the literature [17] to [19] (Table 2.).

We used thermochemical treatments, such as nitro-carburizing and oxy-nitriding to study the effect of microstructure on the wear behaviour of both the simple and hybrid parts. Surface treatment parameters usually applied for 1.2343 steels were selected for the experiments (atmosphere: 50 % nitrogen (4 m³/h), 45 % ammonia (3.7 m³/h) and 5 % carbon dioxide (80 l/h)). The usual consequences of nitriding or nitro-carburizing are increased surface hardness and wear resistance, lower wear coefficient, increased fatigue resistance, ductility, and increased corrosion resistance, which usually result in increased tool life. The additional oxidation of the nitrided layer results in an additional improvement of all the surface characteristics mentioned above. Commercial oxy-nitriding involves nitriding for several hours followed by oxidation in a superheated steam atmosphere.

1.4 Test Methods

1.4.1 Microstructure and Pore Characterization

Both microstructure and pores were characterized on the polished cross-sections. Samples for metallographic examination were etched with Nital and examined with an optical microscope (Zeiss Axio Imager A1).

The transition zones of the hybrid parts were characterized with a scanning electron microscope equipped with EDS (Hitachi 3400).

1.4.2 Hardness Test

The macro and microVickers hardness of the sintered samples and the hardness profiles of the nitrided materials were tested.

We used the Vickers method to measure surface and core hardness based on the MSZ EN ISO 6507-1 standard [22] with KB750 and KB30 hardness testing machines. Surface hardness was tested both before and after heat treatments (HV10). After nitro-carburizing and oxi-nitriding, core hardness and nitriding hardness depth (Nhd) were determined according to the DIN 50190 standard (HV0.3).

1.4.3 Wear Test and Characterization

The wear resistance of specimens after different heat treatments was compared. We analysed the results by calculating the cross-sectional area and the depth of the wear track obtained with a sliding distance of 360 m (60 min), which minimized the influence of surface irregularities.

Ball-on-disk type tribology tests were performed with a UNMT-1 Universal nano & micro surface tester. Zirconium oxide ceramic balls (HV1280; diameter of 5 mm) were used as the sliding part and for each test, a new ball was applied. The sliding speed was 100 mm/s (rev. of 318.47 1/min), and the normal load was 20 N. The tests were repeated three times in each case with the same test conditions (Fig. 1).

a) b)

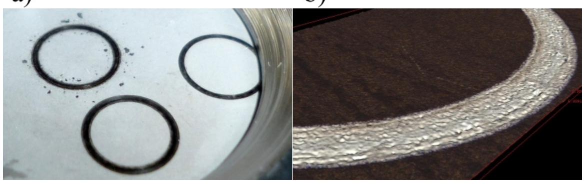

Fig. 1. *The test specimen a) after the wear test, b) confocal microscopic image*

For the analysis the effects of the different heat treatments on wear resistance, we determined the worn area by with series of 2D profile measurements of the wear tracks. The detailed description of the test method and the results of the 3D surface scan (the determination of worn area) can be found in [23]. Authors used a Hobson – Talysurf CLI2000 scanning surface topography instrument for the experiments (contact method, 100 Hz, radius of curvature: 5 µm). First, the centre of the wear track torus was determined, then the profile along two mutually perpendicular diameters was measured (see Fig. 2). The coordinates in a text file were stored, then the worn areas were calculated with the algorithm we developed.

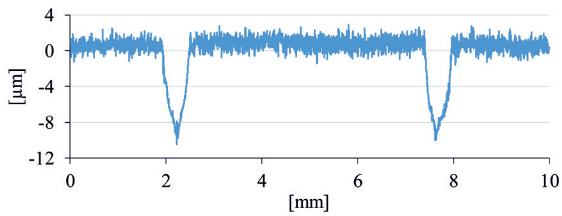

Fig. 2. *The 2D profile of the worn*

2 RESULTS

2.1 Microstructure and Porosity

The samples made with different manufacturing processes show different macroscopic characteristics. The metallographic examination showed that the DMLS material had a layered structure (Fig. 3) and the thickness of the structural layers was around 20 µm to 30 µm, consistent with sintering layer thickness. All layers had a regular wave-like form. In the microstructure of the samples made with the conventional manufacturing process, the direction of rolling is clearly recognizable.

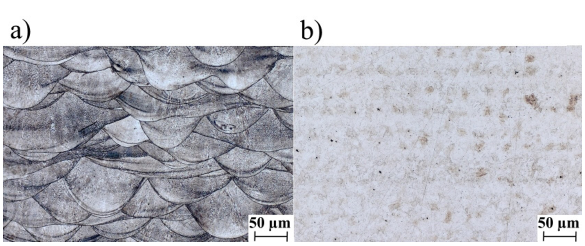

Fig. 3. *The side view of a) the DMLS material (MS1) showing the wavy layers, and b) the conventional W722 steel before heat treatment*

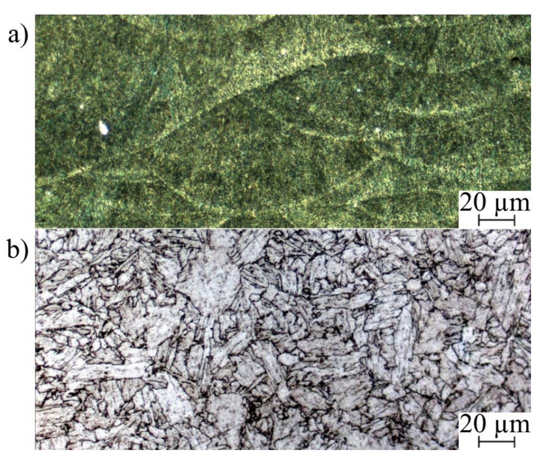

Fig. 4. *Microscopic images of a) age-hardened MS1, and b) W722 (Nital 2 %)*

The microscopic images of the age-hardened MS1 steel clearly show the typical wave-like structure of layers superimposed on each other, which presumably contain precipitations of the nanometre scale. The massive bundles of martensite can be clearly identified in the microstructure of W722. Finely dispersed intermetallic particles composed of the alloying elements can be clearly seen in the grains (Fig. 4).

2.2 Surface Hardness after Age Hardening and Hardening

To analyse the effect of heat treatment in the case of hybrid metals, we used age hardening for W722-MS1 and both age hardening and quenching for the 1.2343-MS1 combination. The results can be seen in Figure 5. Hardness increased significantly as a result of age hardening. Nearly the same hardness was achieved in the case of MS1 (sintered) and W722 (conventional) maraging steels. While the hardness of these two materials was ~590 HV when age-hardened, the heat treatment performed with the same technological parameters softened the 1.2343 steel. More suitable heat treatment (quenching and tempering twice), usually suggested for 1.2343 steel, resulted in the same hardness as that of the sintered MS1.

Nearly the same hardness can be achieved by three-step hardening and age hardening in the case of MS1 (Fig. 5).

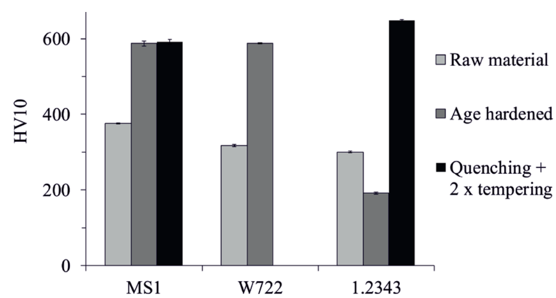

Fig. 5. *Surface hardness of MS1, W722 and 1.2343 materials before and after heat treatment*

This can be explained with the low differences between the temperature of tempering and age hardening, while the duration of the treatment is the same.

2.3 The Effect of Nitriding on the Microstructure of Hybrid Systems

Nitrided and carbonitrided layers typically consist of two areas. The inner (diffusion) region is characterized by the formation of nitride needles. Normal layer thickness ranges from 0.2 mm to 1.5 mm. The outer region that is approximately 5 µm to 30 µm thick is called the compound layer. This non-metal layer consists mainly of γ'-nitrides (Fe_4N), ε-nitrides (Fe_xN) and possibly of carbonitrides ($Fe_xC_yN_z$). Moreover, in alloyed steels nitrides and carbonitrides of the alloy elements are formed. The outer part of the surface layer can be formed as a porous edge, which later improves anti-corrosive properties through oxidation.

In the case of nitro-carburizing both nitrogen and carbon diffusion occur and similarly to nitriding, a compound layer of a few µm containing carbonitrides and an underlying diffusion zone of high nitrogen content is developed.

2.3.1 MS1 – W722 Hybrid Part

MS1 steel powder was sintered directly onto the surface of W722 steel; the hybrid part was then nitrocarburized. In the microscopic images, the parts produced with different technologies are well separated. The boundary between the two materials is not sharp; a ~50 µm-wide transition zone can be seen as a mixture of two materials, tracking the contour of the laser-scanning structure as images in Fig. 6 show.

a)

b)

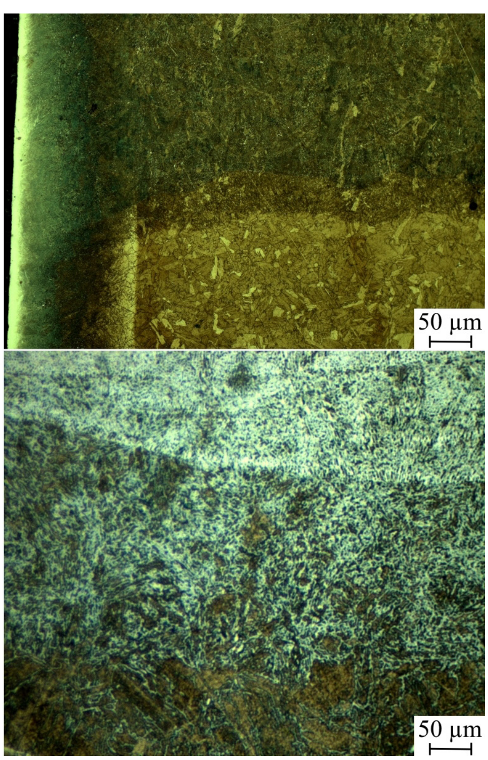

Fig. 6. *The microstructure of maraging steels after nitro-carburizing; a) the hybrid structure of W722 and MS1 steel, and b) the boundary area of the hybrid structure (Nital 2%)*

The thickness of the compound layer is nearly the same in the case of both materials; 13 µm for the MS1 and 12 µm for the W722 part.

Fig. 7 shows microhardness as a function of distance from the surface, i.e. the microhardness profiles obtained for the nitrocarburized (N) and oxy-nitrided (NO) MS1 and W722 steels. The initial part of the curves drops sharply, which indicates the low diffusion depth of C and N.

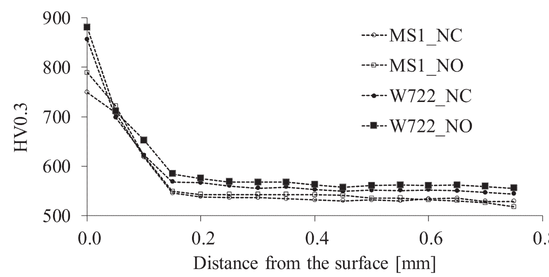

Fig. 7. *The HV0.3 microhardness profile of nitrocarburized (NC) and oxy-nitrided (NO) MS1 and W722 steels*

Surface hardening resulted in lower hardness in the core in each case, compared to that of age-hardened samples (Fig. 8). It can be explained with the higher duration time of the applied thermochemical treatments. On the one hand, during age hardening fine particles, typically coherent or semi-coherent inter-metallics are produced, which efficiently impede the movement of dislocations, and defects of the crystal lattice are created, which harden the material. On the other hand, during nitro-carburizing, the longer duration time leads to the further ripening and growing of non-equilibrium particles, resulting in the formation of incoherent precipitations of larger size and of more equilibrium composition. Altogether, this process, called over-aging, decreases the strength and hardness of the material.

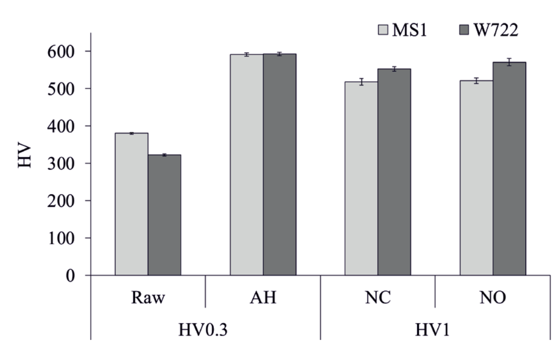

Fig. 8. *Core hardness of MS1 and W722 steels in different heat treatment conditions*

The decrease of hardness due to nitro-carburizing compared to age hardening is higher for MS1 (50HV) than for W722 (30HV). Therefore, over-ageing during nitro-carburizing is stronger in the case of MS1 than in the case of W722, i.e. greater reduction of hardness can be expected in the case of the MS1 steel. Nhd was determined in the cases of both materials and both nitriding processes, resulting in Nhd = 0.13 mm for MS1, and Nhd = 0.12 mm for W722 steel.

2.3.2 MS1 – 1.2343 Hybrid Part

In earlier research [24], authors produced hybrid structures using commercial tool steels of different chemical compositions and heat treatment specifications. Heat treatment was used in the different steps of the manufacturing process, and the residual stresses generated during processing were described based on deformation measurements. The authors concluded that hardening is suggested before sintering in all cases when the DMLS process is applied on a raw material different than that of a maraging type tool steel, and the prescribed surface hardness value of the mould is similar. Otherwise, substantial internal stresses in the material would be generated during the high-temperature heat treatment after construction [24].

In this paper, MS1 steel powder was sintered onto both the untreated and the quenched 1.2343 steel plates. In the case of untreated (raw material) base plates, the hybrid part was first quenched and double tempered, followed by nitro-carburizing. In this case, the hardness of both materials was ~600HV, while in the case of a quenched base plate and a hybrid part subjected only to nitrocarburizing, the hardness of the sintered MS1 material was altogether ~380HV.

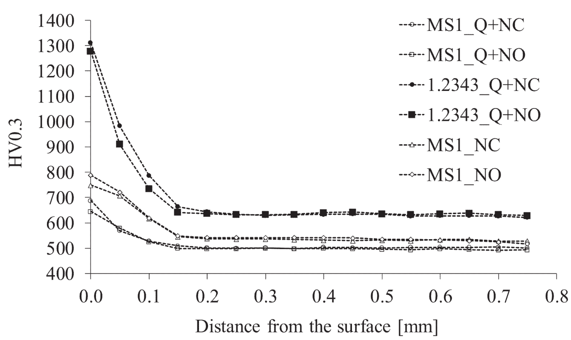

Fig. 9. *HV0.3 microhardness profile of nitrocarburized (NC) and oxy-nitrided (NO) steels*

The nitro-carburizing hardness profile clearly shows that the hardness of the quenched and nitrocarburized 1.2343 material at a distance of 0.05 mm from the surface is ~200HV higher than that of the nitro-carburized MS1 steel (Fig. 9.) because of the higher amount of nitride-forming elements and carbon concentration.

The hardness at a distance of 0.05 mm from the surface of the maraging steel that was quenched and then nitrocarburized was ~200HV lower than that of the MS1 steel subjected only to nitrocarburizing. This can be explained with two phenomena of the over-aging caused by the long holding time of the Q-type heat treatment; (i) the metastable martensite reversion into austenite and (ii) the coarsening of the intermetallic precipitates.

2.4 Transition Zone in Hybrid Structures

The quality of the transition zone in the case of hybrid tool inserts has outstanding importance in terms of operation and lifetime. In order to avoid the wear of the tool locally, the hardness of the transition zone cannot deviate significantly from the homogeneous parts of the tool. In our experiments, MS1-W722 and MS1-1.2343 hybrid samples were tested. Microscopic analysis (Fig. 10) and HV0.3 hardness measurements were carried out on the transition zone and its environment.

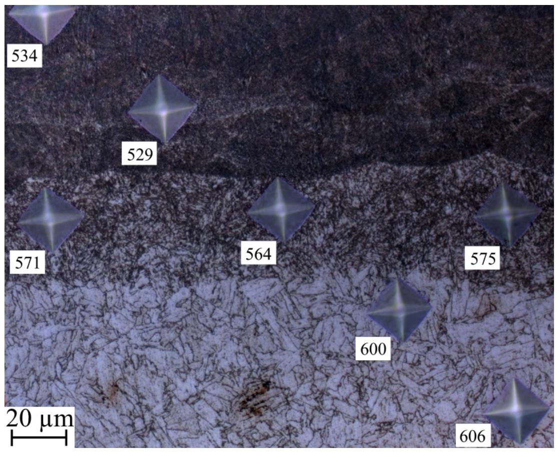

Fig. 10. *Microstructure of the age-hardened MS1-W722 hybrid part (W722 on the bottom, MS1 on the top and the transition layer in the middle) with the traces and resulted values of HV0.3 hardness measurement*

As seen in Fig. 10, the transition zone, which was melted by the laser beam during the sintering, resulted in a finer structure of W722.

Based on our results, the hardness of the transition layer of the MS1-W722 hybrid part after age hardening can be expected between the hardness values of the homogeneous base materials. The hardness of the boundary layer between the MS1 and the 1.2343 in the hybrid structure is slightly lower than that of base materials (Fig. 11).

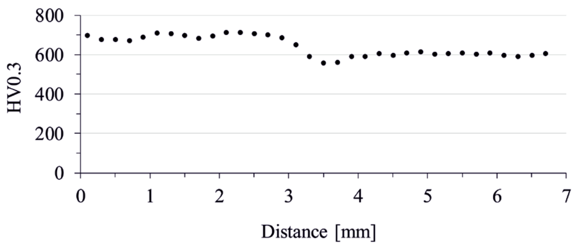

Fig. 11. *HV0.3 hardness measurement in the cross-section of the MS1-1.2343 sample*

Scanning electron microscopy combined with EDS analysis was used to study the transition layer in the MS1-1.2343 hybrid part (Fig. 12).

In the case of this material combination, the transition zone cannot be seen as clearly as in the case of MS1-W722. Element map analysis was carried out, and the Ni and Cr distribution was studied to characterize the transition layer.

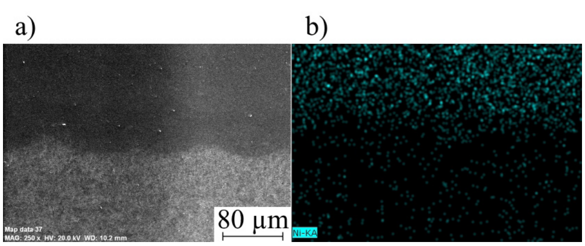

Fig. 12. *a) SEM image (MS1 – top, 1.2343 – bottom) and b) Ni element map of EDS analysis of the MS1-1.2343 hybrid part*

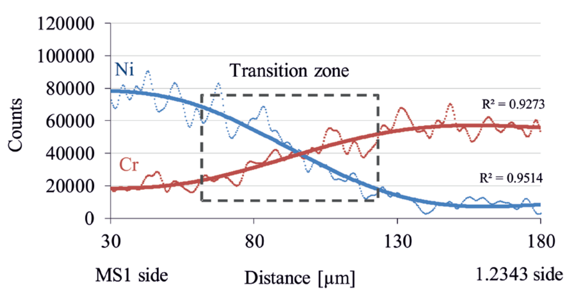

Fig. 13. *Changing the Ni and Cr element concentration from the MS1 side to the 1.2343 material*

The element concentration of Ni and Cr alloying elements in different cross sections from the MS1 side to the 1.2343 side was characterized by image analysis with the use of the element map images (Fig. 13). The values on the y-axis of the diagram correlate with the quantity of the elements in the appropriate cross section. The concentration of the elements changes continuously in the transition layer, which can ensure the correct interfacial properties, but more investigations are needed to verify this.

2.5 Porosity

Polished cross-sectional surfaces of the hybrid parts were examined with an optical microscope, revealing pores of micrometre size on the MS1 side of the part (Fig. 14.). The area-related amount of porosity can be determined in the microscopic image. The 0.32% porosity of the sintered MS1 part decreased after nitro-carburizing to 0.28 %.

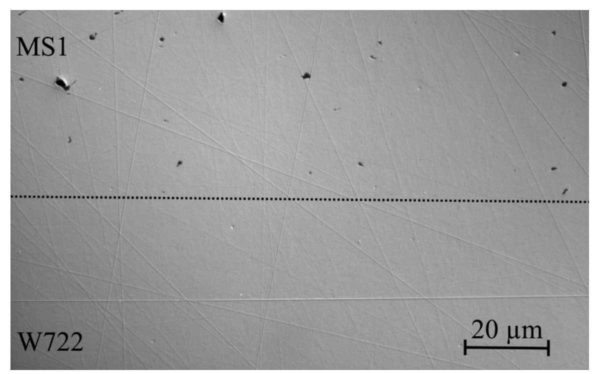

Fig. 14. *Morphology and location of the pores on the polished cross-section of the DMLS material*

2.6 Wear Test

Wear tests were carried out on age-hardened polished MS1 samples with the same normal load and different duration time (10 min, 20 min, 30 min, 60 min, 120 min, 240 min).

To evaluate wear resistance and characterize the time-dependent nature of the wear process, wear track depth, and the cross-sectional area of wear with 2D profile diagrams were determined. Fig. 15 shows these two parameters as a function of time.

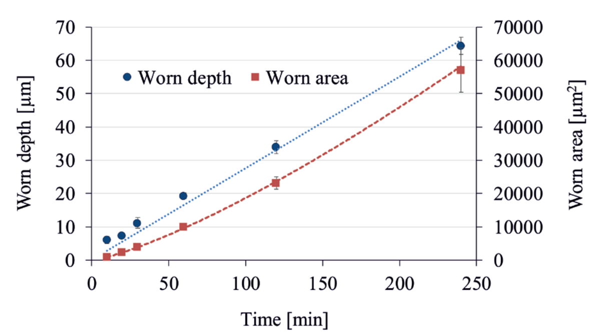

Fig. 15. *Depth of wear track and the worn area as a function of sliding time*

Material loss after an initial burn-in phase is considered to be linear. Taking these results into account, we performed the subsequent tests for a sliding time of 60 min.

Fig. 16. shows the results of the three material grades for different heat treatments.

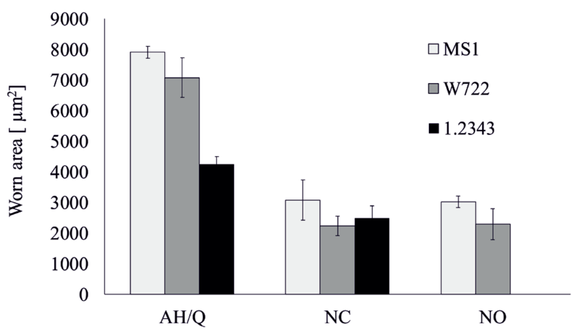

Fig. 16. *The measured values of the worn area of MS1, W722 and 1.2343 steel in age hardened or quenched (AH/Q); nitrocarburized (NC) and oxy-nitrided (NO) conditions*

The worn area decreased significantly after nitro-carburizing and oxy-nitriding, thus providing higher wear resistance. In addition, wear resistance was higher for the W722 material compared to that of the MS1 steel for all the investigated heat treatment conditions.

Compared to the quenched and tempered 1.2343 steel, a worn area twice the size was measured in case of age-hardened MS1 steel, which can clearly be attributed to the higher hardness of conventional steel. The lower wear resistance of the sintered and nitrocarburized MS1 materials compared to that of the nitrocarburized, and Q+2T treated 1.2343 steel can be explained with the presence of micro-porosity, while the cause of the lower hardness of MS1 is over-ageing during nitro-carburizing.

As a result of nitrocarburizing, the wear resistance of MS1 and W722 steels was improved by ~65 % compared to their wear resistance without surface treatment. The wear test results of hybrid model structures composed of differently heat-treated base plate and insert materials suggest that nitriding and nitro-carburizing are effective ways of increasing the wear resistance of hybrid tool inserts.

3 CONCLUSIONS

We used DMLS to produce hybrid structures combining conventional processing technology with rapid prototyping. W722 maraging steel of equivalent chemical composition as the sintering powder and 1.2343 conventional tool steel were selected as base materials, and MS1 maraging steel powder was used for laser sintering. Different heat treatments (age hardening, quenching, quenching with double tempering, nitro-carburizing and oxy-nitriding) were applied after production. We characterized the microstructure, hardness and wear properties of the different steels, and studied the hybrid structures and analysed them from a tribological point of view.

We showed that the different manufacturing processes resulted in different microstructures in the case of laser-sintered and hot-rolled maraging steels, as well as the higher porosity of the laser-sintered material. Comparing the effect of age hardening and quenching on the hardness of the different steel grades, we found that age-hardening results in almost the same hardness as quenching in maraging steels, while the same heat treatment resulted in softening in the case of 1.2343 steel. Quenching produced the same hardness as age hardening in MS1 steel. Heat treatment parameters have a significant impact on the mechanical properties. In the case of hybrid structures, it is necessary to select heat treatments and heat-treating parameters that produce the required properties in both components.

The transition zone of both the MS1-W722 and MS1-1.2343 hybrid parts was studied. EDS analysis showed continuous changing of element concentration in the transition zone. There is no sharp boundary between the two materials in the hybrid part, neither in the microstructure nor in the mechanical properties.

We confirmed experimentally that laser-sintered parts possess higher porosity compared with conventionally made steels, which explains the lower wear resistance of MS1 samples, compared to W722 maraging steel samples made from a conventionally produced steel bar.

The higher wear resistance of 1.2343 steel compared to that of maraging steel can be ascribed to its higher carbon content, and its higher attainable surface hardness. When nitro-carburizing was applied, the worn area, which is an indication of wear resistance, dramatically decreased and nearly the same value was achieved for all steels. The sintered maraging steel showed lower wear resistance than its conventionally produced counterpart did in all cases, which can be explained with the higher porosity as a result of sintering.

Our results indicate that nitro-carburizing provides an effective way to increase the wear performance of hybrid tools.

We showed that if tool steel is used in hybrid constructions requiring quenching to assure the prescribed properties, this heat treatment can produce the same hardness in the MS1 part as age hardening can when high-temperature tempering is applied after quenching.

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