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THE INFLUENCE OF Nb, Ta AND Ti MODIFICATION ON HOT-WORK TOOL-STEEL GRAIN GROWTH DURING AUSTENITIZATION

VPLIV MODIFIKACIJE ORODNEGA JEKLA ZA DELO V VROČEM Z Nb, Ta IN Ti NA RAST ZRN MED AVTENITIZACIJO

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In order to explore the influence of niobium, tantalum and titanium modification on grain growth in high-thermal-conductive hot-work tool steel during austenitisation at high temperatures, three types of modified steels (sample HTCS-130 + 0.06 *w*/% Nb, sample HTCS-130 + 0.03 *w*/% Ta, sample HTCS-130 + 0.006 *w*/% Ti) based on reference (sample HTCS-130 – 0) were prepared. The effect of different austenitisation temperatures (1030, 1060, 1080 and 1100) °C on hardness after quenching and grain size were investigated. The results show that there is a positive effect on the mechanical properties and a decreased grain-growth effect in the modified steel samples. The precipitation behaviour of the carbides was also investigated with electron microscopy. The Mo-W carbides were relatively weak at retaining grain size, but their pinning effect was increased with the incorporation of other carbide-forming elements like Nb and Ta. MC-type carbides on the grain boundary were effective at grain-boundary pinning. Nb even further increased the resistance by forming NbC carbides. The addition of Ti, on the other hand, proved to be ineffective due to the intergranular precipitation of the formed carbonitrides.

Keywords: hot-work tool steel, microalloying, quenching, grain growth, carbides

Z namenom preiskave vpliva modifikacije Nb, Ta, in Ti na visoko toplotno prevodno orodno jeklo za delo v vročem med procesom avstenitizacije pri povišanih temperaturah, smo na osnovi referenčnega materiala (vzorec 0 – HTCS-130), razvili tri različna nova modificirana jekla (vzorec HTCS-130 + 0,06 *w*/% Nb, vzorec HTCS-130 + 0,03 *w*/% Ta, vzorec HTCS-130 + 0,006 *w*/% Ti). Raziskali smo vpliv različnih temperatur avstenitizacije (1030, 1060, 1080 in 1100) °C na trdoto po kaljenju in velikost zrn. Rezultati kažejo pozitiven vpliv mikrolegirnih elementov na povišanje mehanskih lastnosti in zaviranje rasti avstenitnih zrn. S pomočjo elektronske mikroskopije smo raziskali izločanje karbidov. Mo-W karbidi niso imeli večjega vpliva na zaviranje rasti kristalnih zrn, z dodatkom Nb in Ta pa se je rast zrn bistveno upočasnila. MC tip karbidov je bil učinkovit pri zaviranju rasti zrn. Dodatek Nb je dodatno zadrževal rast zrn s tvorbo NbC karbidov. Dodatek Ti se je izkazal za neučinkovitega, zaradi intergranularnega izločanja titanovih karbonitridov.

Ključne besede: orodno jeklo za delo v vročem, mikrolegiranje, kaljenje, rast zrn, karbidi

1 INTRODUCTION

Hot-work tool steels are generally used in die casting, extrusion, and hot-forming processes. These processes mean exposure to complex thermal and mechanical loads, and even chemical degradation. Due to these reasons, the material sometimes has to withstand severe conditions during the working process.[1–3] The main properties that a hot-working tool steel has to possess are connected with tempering resistance, increased strength and high hardness, fatigue and thermal shock resistance, impact toughness, sliding wear resistance, etc.[4]

One of the properties that is also important is thermal conductivity. It represents the ability of the material to transfer heat during and after loading cycles under increased temperature.[2,5,6] Increasing the thermal conductivity of steel can have an influence on total time of the production cycle. This means that the process is more cost efficient and the productivity of the process is higher.[2,6,7] The value of the thermal conductivity is most affected by the material's chemical composition. Elements such as Cr and V, which are commonly used in hot-work tool steels, in combination with other elements decrease the value of the thermal conductivity. Lowering the values of these elements increases the materials' ability to transfer heat.[5,6] New types of steel with increased thermal conductivity values up to 60 W·(mK)$^{-1}$ have been developed, to fulfil these requirements.[2,8,9]

The final material properties depend on the chemical composition, production process and heat-treatment process.[1,4] During the heat treatment the final microstructure and mechanical properties are formed that are crucial for final the durability and lifetime of the tool.[4,10,11] The typical temperature range of heat treatment for tool steels type AISI H11 or AISI H13 is in the range from 1000 °C to 1050 °C. The material is heated to the austenitisation temperature and rapidly cooled – quenched using a cooling medium, most commonly oil or nitrogen, and air for

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hot-work tool steels.[1,13–16] During heat treatment grain growth can occur, due to the high austenitisation temperatures. The increase of the austenitisation temperature or the holding time can increase the grain size from fine to coarse, resulting in decreased material toughness. By adding elements such as W to the chemical composition, higher austenitisation temperatures must be chosen to achieve the optimum mechanical properties. This means that the temperatures during austenitisation rise above 1050 °C and can cause excessive grain growth.[17]

Grain growth can be affected by adding alloying elements such as Nb, Ta or Ti to modify the base chemical composition of the steel. These elements form carbides and carbonitrides that supress the grain growth.[18–22] In addition to grain-growth retention, the addition of these elements can increase the peak annealing temperature, tempering resistance and enhance the mechanical properties in general.[23–25]

The steel's hardenability is increased by adding alloying elements such as C, Mo, Mn, Cr and Ni. Other elements can also be added for additional precipitation hardening, such as W, Nb, Ti and Ta.[26–28] Many types of carbides can be formed in the matrix of hot-working tool steels, for example M_3C, $M_{23}C_6$, M_7C_3, M_6C, M_2C and MC. Carbide precipitation influences the wear and mechanical properties, but it can decrease other properties, for example thermal conductivity.[29,30]

This investigation focuses on a high-thermal-conductive tool steel HTCS-130, with the objective being to determine the effect of Nb, Ta and Ti on grain growth at elevated temperatures. The intention was to achieve a finer grain size and increased hardness after quenching and tempering. The microstructure was investigated and the role of microalloying elements was analysed.

2 EXPERIMENTAL PART

Experimental charges of HTCS-130 steel were remelted in a vacuum induction-melting furnace under 300 mbar of argon. In total four charges of 8 kg were cast into cast-iron moulds to form ingots of dimensions (60 × 60 × 400) mm. The first charge of HTCS-130 was made without any additional alloys. The second charge had an addition of 0.06 *w*/% Nb, the third charge had an addition of 0.03 *w*/% Ta and the fourth charge had an addition of 0.006 *w*/% Ti. After casting the ingots were air cooled to room temperature.

The chemical analysis of the steel samples was made using an ELTRA CS-800 (C and S) and an ELTRA ON-900 (N and O), and using an optical emission spectrometer with inductively coupled plasma ICP-OES Agilent 720 (other elements).

The ingots were annealed at 720 °C for 2 h for stress relieving. The ingots were then homogenized at 1200 °C for 1 h and hot rolled to billets of dimensions 40 mm × 40 mm. After hot rolling the billets were cooled down slowly in air to room temperature. The billets were then additionally heated to 1200 °C and hot forged into square bars of dimensions 18 mm × 18 mm. The bars were then cooled slowly in air to room temperature. After cooling to room temperature the samples were soft annealed at 770 °C for 1.5 h in a vacuum hardening furnace (Ipsen Turbo XL).

Samples were taken from all operations: casting, hot rolling, hot forging, and soft annealing. Additional analyses were conducted on the samples. The samples after heat treatment were machined to remove the decarburised layer formed during exposure to high temperature and no protective atmosphere.

Then the samples were cut to dimensions (18 × 18 × 60) mm. The hardening process was made in an air-atmosphere electric laboratory furnace followed by oil quenching. The temperature was measured in dummy samples with type-K thermocouples in drilled holes. The samples were put into a room-temperature resistance furnace, heated to 650 °C in 1 h, and held for 20 min, then heated to 850 °C in 30 min and again held for 20 min and then heated to the quenching temperature in 45 min and held for 20 min. The quenching temperatures were (1030, 1060, 1080 and 1100) °C. The oil temperature was 60 °C. After oil quenching the samples were tempered in a vacuum hardening furnace under a protective atmosphere at temperatures (540, 580, 600, 620 and 640) °C with a holding time of 2.5 h. The samples were milled, removing 2 mm of the surface layer, to avoid the influence of decarburisation.

The metallographic samples were ground, polished and etched with Nital 5%. The microstructure was analysed with a light microscope (Olympus DP70) and a scanning electron microscope (Thermo Scientific Quattro S). Furthermore, microchemical analysis with electron dispersive x-ray spectroscopy (EDS) was also performed. The grain size was determined according to ASTM E112. Hardness values were measured on all the investigated samples using the Vickers hardness method, HV10 (Instron Tukon 2100B).

3 RESULTS AND DISCUSSION

After soft annealing at 770 °C the hardness of all the samples was below 280 HV. The samples were ground before the quenching process. The chemical analysis of the samples is given in **Table 1**. The investigated samples were alloyed with C, W and Mo. The absence of some typical alloying elements in hot-work tool steels, such as Cr, increases the thermal conductivity.[22,29]

The investigated samples were quenched from temperatures of (1030, 1060, 1080 and 1100) °C with 20 min of holding time. The samples were heated without a protective atmosphere, which resulted in significant scaling and decarburisation. After the hot forming and final heat treatment the decarburised layer was approximately 1.5 mm thick. The pronounced decarburisation occurred due to the absence of Si and Cr in the steel.[1,4,31]

Table 1: Chemical compositions of the experimental charges (*w*/%)

Sample	C	Si	Ni	Mo	W	Nb	Ta	Ti	N	Fe
HTCS-130 – 0	0.32	0.04	0.03	3.2	1.7	–	–	–	0.001	Bal.
HTCS-130 + Nb	0.32	0.05	0.03	3.2	1.7	0.06	–	–	0.001	Bal.
HTCS-130 + Ta	0.32	0.05	0.03	3.2	1.7	–	0.03	–	0.001	Bal.
HTCS-130 + Ti	0.32	0.04	0.03	3.2	1.7	–	–	0.006	0.001	Bal.

As mentioned before, the decarburised layer was removed by machining, so it would not influence the results. The hardness was measured after oil quenching and further also after each tempering. A quenching matrix was made to obtain the hardness properties formed using different quenching and tempering conditions.

Table 2: Hardness measurements after quenching in oil (HV10)

Sample hardness (HV10)	Temperature (°C)			
	1030	1060	1080	1100
HTCS-130 – 0	425	423	484	473
HTCS-130 + Nb	417	459	448	502
HTCS-130 + Ta	414	468	455	475
HTCS-130 + Ti	446	422	461	485

The effect of different quenching temperatures on the hardness after quenching is presented in **Table 2**. In general, the hardness values increased with increasing temperature. All the samples achieved hardnesses between 414 HV10 and 502 HV10. The lowest hardness of 414 HV10 was obtained for the Ta-modified sample (HTCS-130 + Ta) quenched from 1030 °C, while the highest value belongs to the Nb modified sample (HTCS-130 + Nb), quenched from 1100 °C. The unmodified sample HTCS-130 achieved the highest hardness at 1080 °C, i.e., a value of 484 HV10, while all the modified samples achieved the highest hardness at 1100 °C. Sample HTCS-130 + Nb value of 502 HV10, sample HTCS-130 + Ta – value of 475 HV 10 and sample HTCS-130 + Ti value of 485 HV10. Based on the results, the highest hardness values are achieved in the temperature range 1080 °C to 1100 °C. The effects of microalloying additions were not straightforward. The added elements Nb, Ta and Ti should have a positive effect on the mechanical properties and also on the grain size. From the results it can be concluded that the highest hardness formed at 1030 °C was 446 HV10 in the sample with the Ti addition (HTCS-130 + Ti), but Nb and Ta had much lower values of 417 HV10 and 414 HV10. Quenching from 1060 °C resulted in lower hardness in the Ti-alloyed sample (422 HV10) and much higher in the Nb and Ta samples, i.e., 459 HV10 and 468 HV10, respectively, while the unmodified sample had 423 HV10. At 1080 °C the hardness of the Ti-alloyed sample increased (461 HV10), while it decreased for the Nb- and Ta-modified samples (448 HV10 and 455 HV10). The highest quenching temperature yielded a lower hardness for the unmodified and Ta-modified samples, 473 HV10 and 475 HV10, while it resulted in higher values for the Nb- and Ti-modified samples, 502 HV10 and 485 HV10, respectively.

The microstructure of the quenched samples consisted of bainite and martensite, as shown in **Figure 1**. Martensite is slightly more resistant to the etchant, so it remains less etched than bainite.

While the microstructures of all the samples were more-or-less the same, i.e., bainite and martensite, the grain size varied. Primary carbides were not detected by light microscopy. The scanning electron microscope (SEM) analysis revealed carbide stringers along the grain boundaries and undissolved carbides in the matrix in all

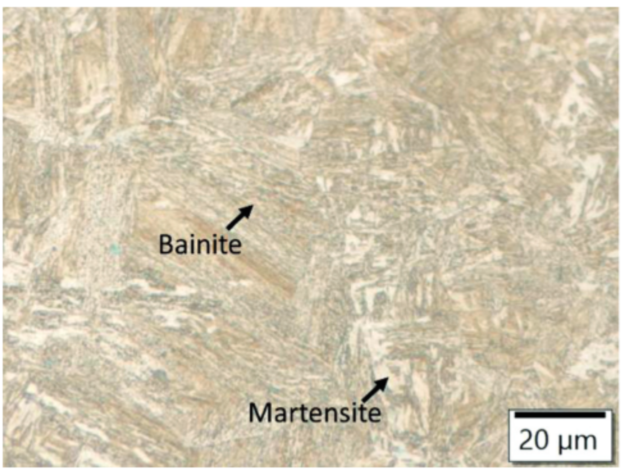

Figure 1: Typical microstructure of the HTCS-130 – 0 steel samples consisting of bainite and martensite (HTCS-130 – 0, quenched from 1060 °C)

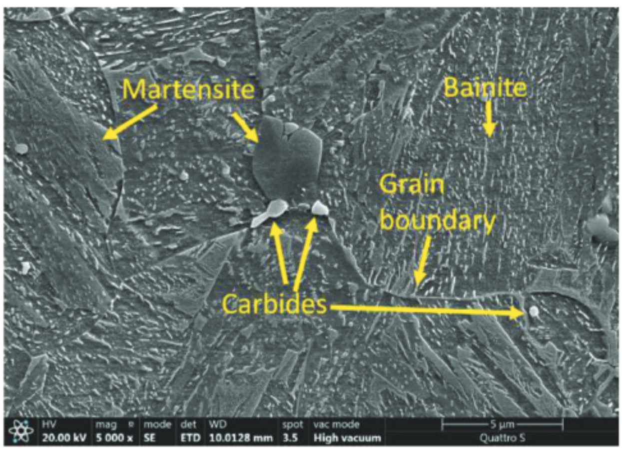

Figure 2: Typical microstructure of the HTCS-130 – 0, quenched from 1030 °C, where martensite and bainite are visible, along with secondary carbides on the grain boundaries and smaller undissolved carbides in the matrix

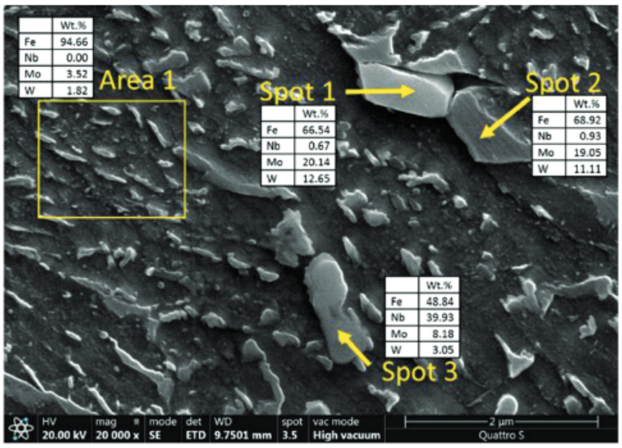

Figure 3: HTCS-130 + Nb quenched from 1060 °C

samples, as seen in **Figure 2** (unmodified HTCS-130 – 0 sample, quenched from 1030 °C). The SEM analysis clearly reveals that the bainite contains carbides, while the martensitic phase is carbide free.

The EDS analysis revealed that the carbides are rich in Mo and W. While all samples contained the Mo-W carbides, the modifications affected their composition and even resulted in the formation of different carbides. Nb-modified samples have Nb carbides and Mo-W carbides that also contain Nb. The level of Nb in the Mo-W carbides is low, but it was detected as seen in **Figure 3**.

Ta carbides were not found in the Ta-modified samples, instead the Ta was incorporated in the Mo-W carbides (**Figure 4**). In fact the concentration of Ta in the carbides was higher than the Nb content in the Nb-modified samples.

The addition of Ti resulted in the formation of Ti carbides that were found in the matrix and not at the grain boundaries. Ti was not incorporated into the Mo-W carbides, as seen in **Figure 5**.

Based on all the hardness measurements, 1080 °C is the most suitable quenching temperature, as all the hardness values exceed 440 HV10, while still avoiding abnormal grain growth. Soaking the material at high temperatures causes grain growth that is deleterious to the mechanical properties. The effect of grain growth was further investigated. Temperatures from 1080 °C to 1100 °C resulted in increased hardness and tempering resistance. Heat treating at higher temperatures can result in grain growth, whereas a fine-grained material is desired to achieve better mechanical properties. The dilemma therefore arises, whether to quench from higher temperatures to ensure a better dissolution of carbides and higher martensite hardness, or to quench from lower temperatures to prevent grain growth at the expense of hardness after quenching.

The addition of micro-alloying elements was predicted to have a positive effect on the average grain size, i.e., less grain growth will occur. Nb and Ti are commonly added to micro-alloyed steel grades to refine the average grain size.[18–20,23,25,26] Ta can also have a positive effect and it promotes the formation of MC carbides, which can have a positive effect on a combination of formed mechanical properties and thermal conductivity.[5,6,22,32] The influence of micro-alloying additions on grain growth was investigated with high-temperature annealing. The grain size results of investigated samples are shown in **Table 3**.

At 1030 °C all the samples were evaluated with an average grain size of ASTM 7. The effects of temperature and alloying elements on average grain size can be seen from **Table 3**.

The process parameters that are most commonly used in industrial practice are at 1080 °C, but above 1030 °C the grain size grows rapidly. At 1060 °C it was evaluated with grade 5. Usually, hot-working tool steels have a grain size of 7 (30 μm) to 8 (22 μm), and in certain cases 6 (45 μm). A bigger grain size has a negative effect on impact toughness. At 1080 °C the average grain size evaluation of sample HTCS-130 – 0 was evaluated with grade 4.5 (78 μm), which can lead to low toughness. Even worse, the grain size at 1100 °C was 4 (90 μm).

The unmodified HTCS-130 – 0 samples (**Figure 6**) exhibited rapid grain growth at temperatures higher than

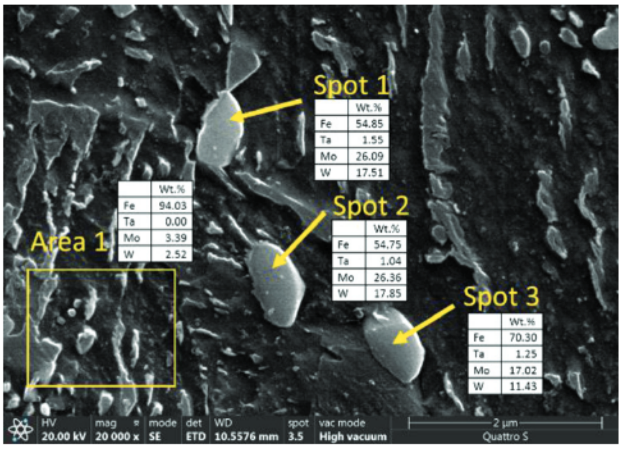

Figure 4: HTCS-130 + Ta, quenched from 1060 °C

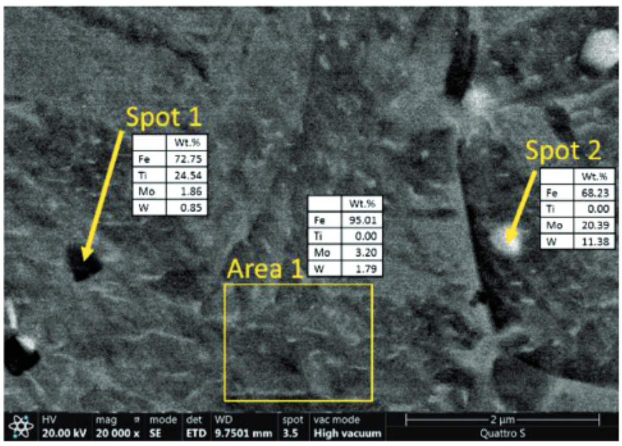

Figure 5: HTCS-130 + Ti, quenched from 1060 °C

Table 3: Grain size evaluation ASTM E112

Temperature (°C)	HTCS-130 – 0 (µm / ASTM)	HTCS-130 + Nb (µm / ASTM)	HTCS-130 + Ta (µm / ASTM)	HTCS-130 + Ti (µm / ASTM)
1030	30 / 7.0	30 / 7.0	30 / 7.0	30 / 7.0
1060	65 / 5.0	30 / 7.0	30 / 7.0	45 / 6.0
1080	78 / 4.5	30 / 7.0	38 / 6.5	65 / 5.0
1100	90 / 4.0	55 / 5.5	65 / 5.0	78 / 4.5

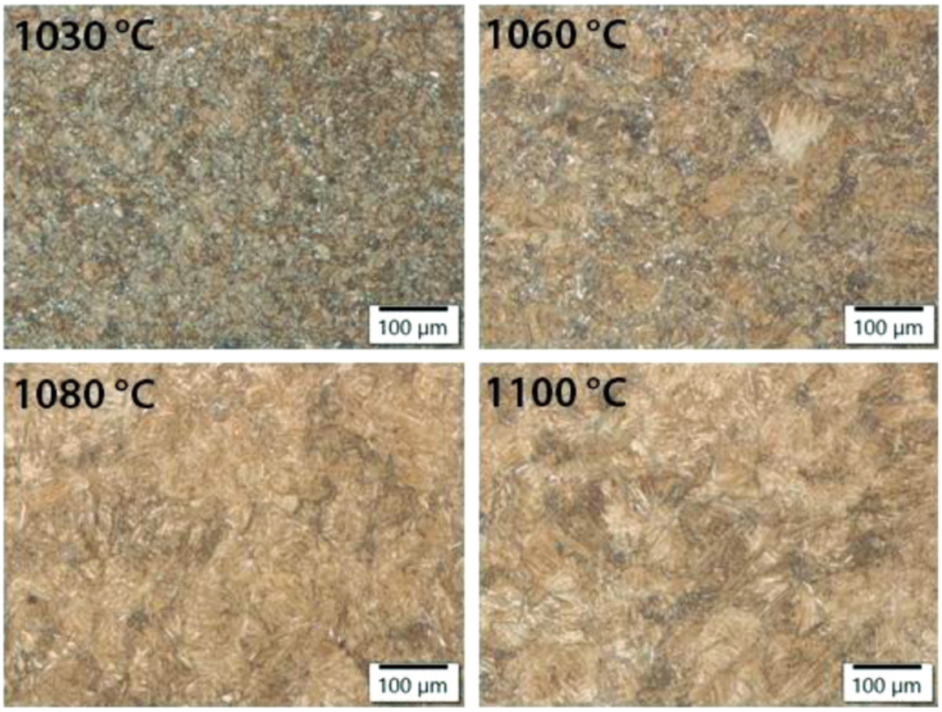

Figure 6: Etched microstructure of sample HTCS-130 – 0, quenched from 1030 °C, 1060 °C, 1080 °C and 1100 °C

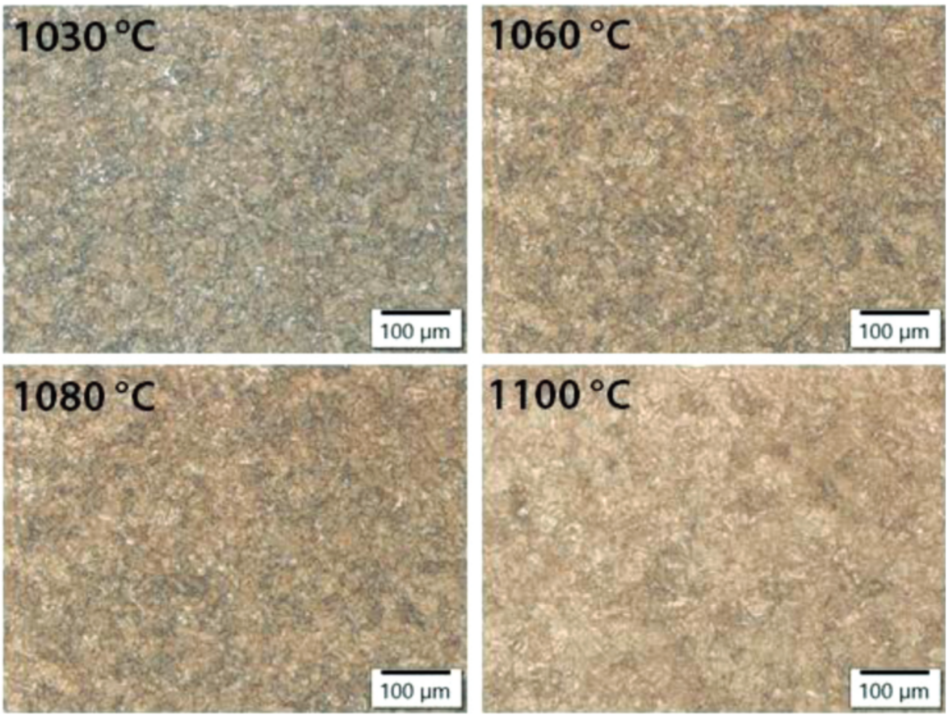

Figure 7: Etched microstructure of sample HTCS-130 + Nb, quenched from 1030 °C, 1060 °C, 1080 °C and 1100 °C

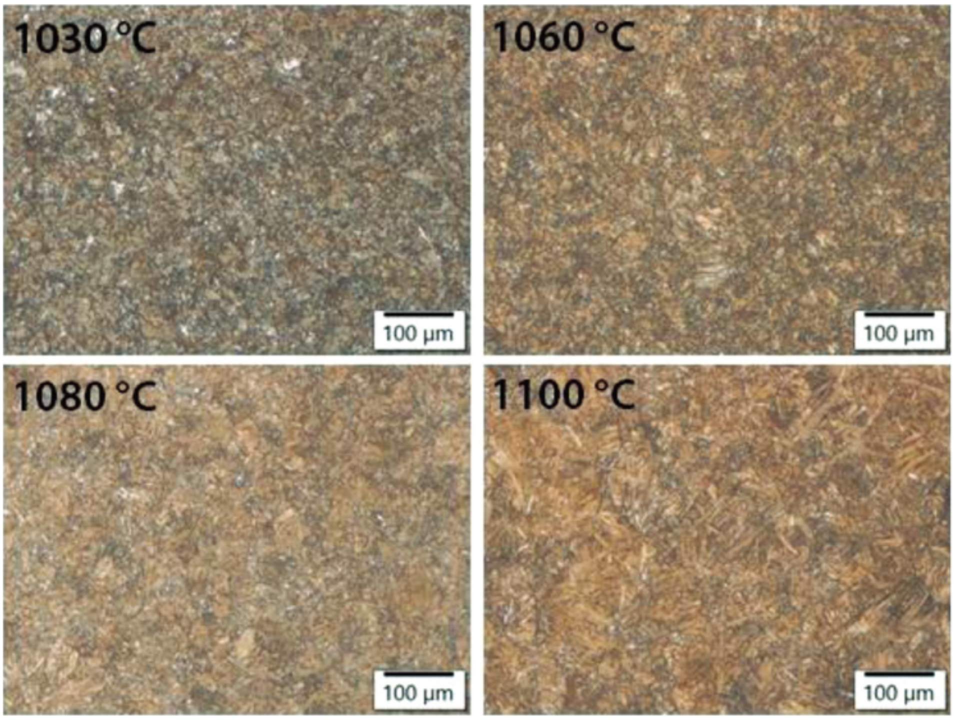

Figure 8: Etched microstructure of sample HTCS-130 + Ta, quenched from 1030 °C, 1060 °C, 1080 °C and 1100 °C

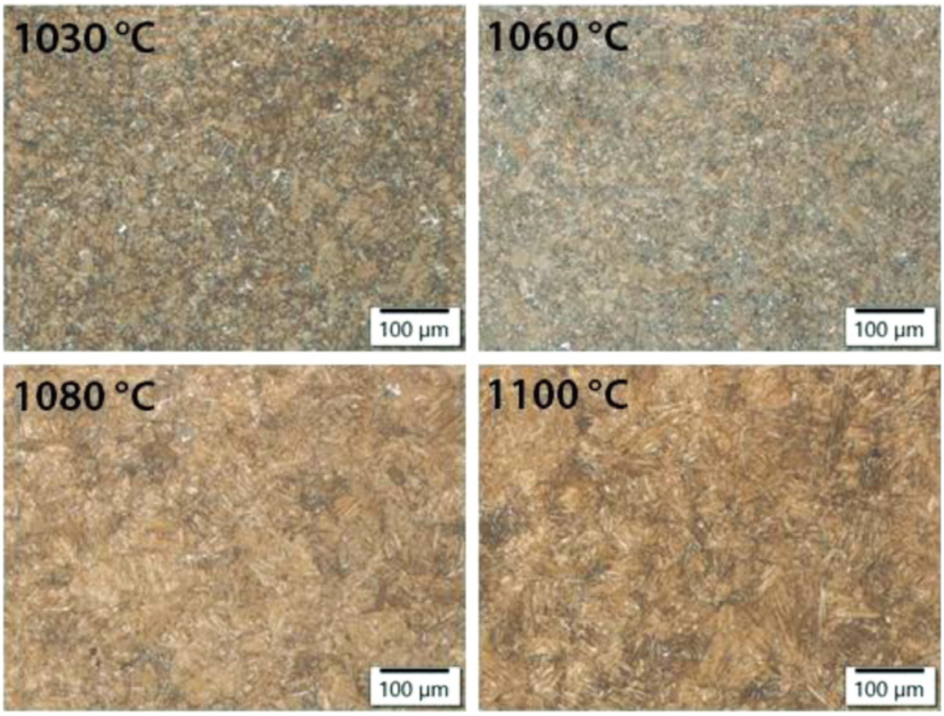

Figure 9: Etched microstructure of sample HTCS-130 + Ti, quenched from 1030 °C, 1060 °C, 1080 °C and 1100 °C

1030 °C. This occurs due to the lack of precipitates on the grain boundaries that pin the crystal grains and thereby retard the grain growth. However, the grain growth is still slightly slower than in unalloyed steels due to the solute-drag effect. SEM investigations revealed some W, Mo carbides along the grain boundaries, but they were apparently not able to have a large effect. Although the grain size at 1030 °C was around 30 μm for all samples, the differences appeared at 1060 °C and increased further at 1080 °C, while 1100 °C proved to be deleterious for grain size in all samples (**Table 4**).

Nb proved to be the most efficient grain refiner, as it held the grain size at 30 μm up to 1080 °C (**Figure 7**), while 1100 °C showed only minor grain growth to

55 µm, compared to 90 µm in HTCS-130 – 0 (**Table 3**). This grain growth resistance is attributed to NbC-type carbides and the incorporation of Nb into the Mo-W carbides.

Ta has a slightly weaker grain-stabilizing effect than Nb, it held the grain size at 30 µm up to 1060 °C (**Figure 8** and **Table 3**). However, the grain growth at 1080 °C and 1100 °C was minor as the grain sizes were 38 µm and 65 µm, respectively. Here the grain-boundary pinning effect is attributed mainly to the stabilization of Mo-W carbides by Ta incorporation, as there are no TaC type carbides in the samples.

Ti surprisingly provided the worst grain size retention, as it proved to be only slightly better than the unmodified sample. Significant grain growth occurred at only 1060 °C (**Figure 9** and **Table 3**). The weak grain-boundary pinning effect of Ti precipitates is attributed to the intragranular distribution. Precipitates, in our case carbides, only function as grain refiners when located on grain boundaries. SEM analysis, however, revealed an intragranular distribution of Ti(C,N)-type precipitates, while Ti was not incorporated into the Mo-W carbides and did not affect the stability. The ineffectiveness of Ti during grain-growth retention can also be linked to low nitrogen values and the low potential of forming TiN.

4 CONCLUSIONS

The addition of Nb, Ta and Ti to HTCS-130 steel resulted in increased grain-growth resistance during high-temperature annealing. Higher austenitising temperatures can result in higher hardness values after quenching. However, they can also result in grain growth, which is deleterious to mechanical properties. Mo-W carbides are relatively weak at retaining grain size, but their pinning effect is increased with the incorporation of other carbide-forming elements like Nb and Ta. MC-type carbides are even more effective at grain-boundary pinning, but must be precipitated at the grain boundary to achieve the effect. Nb and Ta are both dissolved within the Mo-W carbides and increase the grain-growth resistance (up to 1080 °C). Nb even further increases the resistance by forming NbC carbides. Ti, on the other hand, was ineffective due to the intragranular precipitation of the carbonitrides.

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