

## **CHAPTER 4 – RESULTS AND DISCUSSION**

This chapter describes the influence of various heat treatment parameters on the microstructure and hardness of two heats of medium-carbon AISI 420 martensitic stainless steel. The metallographic investigation characterised each heat treated sample by reporting the general microstructure, carbide density, average carbide diameter, ASTM grain size and the percentage retained austenite. Additional data is presented in Appendix A to C. Representative micrographs are shown in this chapter, with the remaining microstructures displayed in Appendix E. The type of carbide present in the as-quenched microstructures was identified using X-ray diffraction techniques and the hardness of each sample was recorded as the average of five measurements (with the 95% confidence interval calculated and reported in each case). Thermodynamic predictions using the CALPHAD™ model are reported for austenitising temperatures between 1075°C and 1175°C.

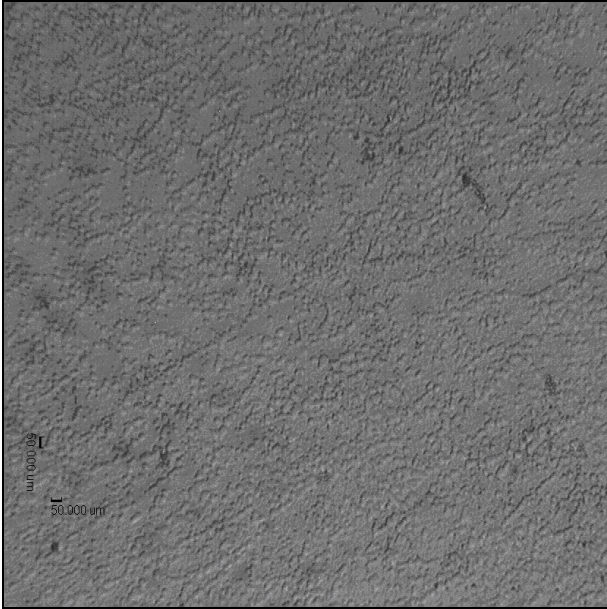
### **1. AS-RECEIVED SAMPLES**

The two heats of medium-carbon AISI 420 material were supplied by Columbus Stainless in the spheroidise annealed condition. The measured as-received hardness was 209±7 HV for HEAT 1 and 195±4 HV for HEAT 2. As shown in Figures 4.1 to 4.3, the microstructures of both steels consist of coarse, globular carbides in a ferrite matrix.

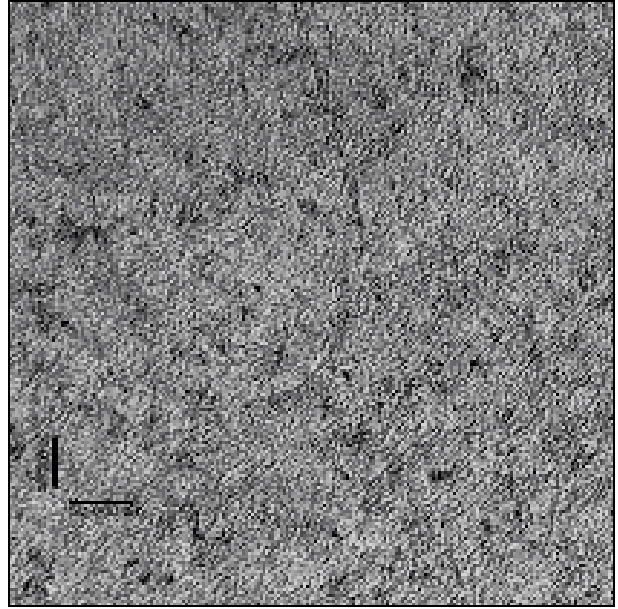
De Andrés *et al*<sup>16</sup> reported that the only carbide present in the spheroidise annealed microstructure of AISI 420 is  $M_{23}C_6$ , however, according to Bjarbo *et al*<sup>11</sup> steels with more than 0.2% carbon and 12 to 13% chromium contain  $M_3C$ ,  $M_7C_3$  and  $M_{23}C_6$  carbides. The precipitation of the carbides is reported to be dependent on time with  $M_3C$  precipitating first, followed by  $M_7C_3$  and then  $M_{23}C_6$ . In this investigation only  $M_{23}C_6$  carbides were identified, with M consisting mainly of iron and chromium.

The higher chromium and molybdenum contents of HEAT 1 result in a higher volume fraction of carbides compared to HEAT 2. The presence of a high volume fraction of carbides is likely to affect the austenitising treatment of both heats. According to the available literature<sup>24</sup>, chromium-rich  $M_{23}C_6$  carbides dissolve in the 950°C to 1050°C temperature range, whereas  $M_7C_3$  carbides dissolve in the 1050°C to 1150°C

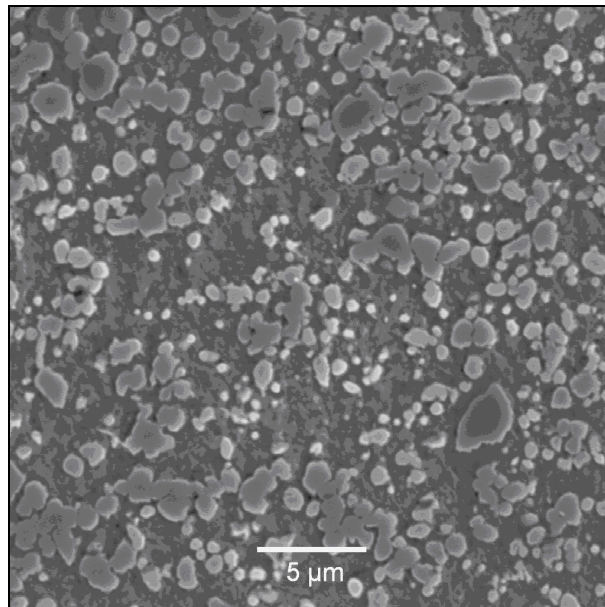
temperature range. A higher austenitising temperature therefore causes more carbides to dissolve. This raises the alloy content of the austenite and depresses the martensite transformation range, increasing the likelihood of retained austenite after quenching.



**Figure 4.1:** The as-received microstructure of HEAT 1, consisting of coarse  $M_{23}C_6$  carbides in a ferrite matrix (hardness:  $209 \pm 7$  HV). (Magnification: 50x).



**Figure 4.2:** The as-received microstructure of HEAT 2, consisting of coarse  $M_{23}C_6$  carbides in a ferrite matrix (hardness:  $195 \pm 4$  HV). (Magnification: 100x).



**Figure 4.3:** Scanning electron micrograph of the as-received microstructure of HEAT 2. The microstructure consists of coarse, globular  $M_{23}C_6$  carbides in a ferrite matrix.

## **2. THE EFFECT OF AUSTENITISING TEMPERATURE ON THE AS-QUENCHED MICROSTRUCTURE AND PROPERTIES OF HEATS 1 AND 2**

When alloying elements dissolve in the steel at high temperatures, the martensite transformation temperatures are depressed. This is illustrated by the equation (4.1), which illustrates the effect of various alloying elements on the  $M_s$  (martensitic start) temperature of 12% chromium steels (all alloy contents in weight percentage)<sup>2</sup>.

$$M_s(^\circ\text{C}) = 500 - 333C - 34Mn - 35V - 20Cr - 17Ni - 11Mo - 10Cu - 5W - 15Co + 30Al \dots(4.1)$$

Equation (4.1) yields predicted martensite start temperatures of 21 °C for HEAT 1 and 46 °C for HEAT 2. These temperatures are close to ambient, suggesting that the martensite transformation is unlikely to go to completion (unless sub-zero treated) if all the alloying elements are in solution in the two stainless steel grades examined. Since the carbides in AISI 420 increasingly dissolve with an increase in austenitising temperature, the martensite start temperature is expected to decrease with higher austenitising temperatures. As shown in Chapter 1, the risk of retained austenite therefore increases with an increase in austenitising temperature. The presence of additional molybdenum in HEAT 1 depresses the martensite start temperature to well below that of HEAT 2, predicting a higher risk of retained austenite after quenching to room temperature.

Table 4.1 displays a summary of the results obtained in this investigation after various austenitising heat treatments. These results are considered in more detail below.

Tavares *et al*<sup>17</sup> reported that the austenitising treatment of AISI 420 should be performed in the temperature range of 980 °C to 1100 °C. Based on a preliminary investigation (not included in this report), a slightly higher austenitising temperature range of 1000 °C to 1200 °C was selected for the purpose of this investigation. Samples of both heats were austenitised at 1000 °C, 1050 °C, 1075 °C, 1100 °C, 1130 °C, 1150 °C, 1175 °C and 1200 °C for 15 minutes. As small samples were used and enough time was allowed for the sample temperature to equalise in the furnace, a 15 minute soaking time was considered to be sufficient for complete austenitising. This was confirmed as part of an unpublished preliminary investigation into the effect of soaking time on as-quenched hardness.

**Table 4.1:** A summary of the results obtained for HEAT 1 and HEAT 2 after various austenitising heat treatments.

**HEAT 1**

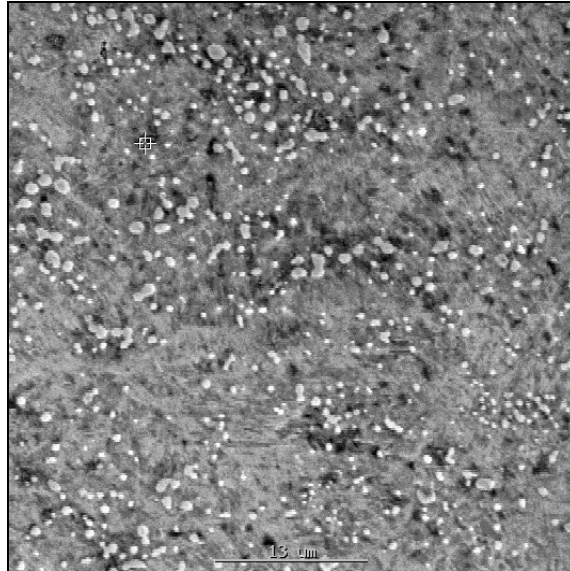
Austenitising temperature	Hardness (HV)	Observed microstructure	% Retained austenite	Carbide density per mm <sup>2</sup>
1000°C	664	Martensite, retained austenite and carbides	4	181
1050°C	678	Martensite, retained austenite and carbides	5	131
1075°C	684	Martensite, retained austenite and carbides	15	87
1100°C	653	Martensite, retained austenite and carbides	23	84
1130°C	474	Martensite, retained austenite and carbides	25	81
1150°C	308	Martensite and retained austenite	27	43
1175°C	279	Martensite and retained austenite	29	0
1200°C	270	Martensite and retained austenite	33	0

**HEAT 2**

Austenitising temperature	Hardness (HV)	Observed microstructure	% Retained austenite	Carbide density per mm <sup>2</sup>
1000°C	639	Martensite, retained austenite and carbides	4	227
1050°C	665	Martensite, retained austenite and carbides	6	184
1075°C	674	Martensite, retained austenite and carbides	10	117
1100°C	639	Martensite, retained austenite and carbides	12	63
1130°C	620	Martensite, retained austenite and carbides	15	32
1150°C	609	Martensite and retained austenite	17	14
1175°C	488	Martensite and retained austenite	21	0
1200°C	459	Martensite and retained austenite	24	0

In order to verify the results reported in literature, HEATS 1 and 2 were austenitised at 1000°C for 15 minutes, a temperature too low to dissolve significant amounts of  $M_{23}C_6$ . As only a small percentage of carbides goes into solution during heat treatment, very little retained austenite is expected after quenching. This was confirmed by XRD analysis which shows that 4% retained austenite was present in both HEATS 1 and 2 after austenitising at 1000°C (as shown in Table 4.1). The as-quenched microstructure, shown in Figure 4.4, consists of coarse, globular carbides in a fine martensitic matrix. The shape and distribution of the carbide particles suggest that they are undissolved precipitates (from the spheroidise annealing treatment), rather than reprecipitated carbides. The carbide density in HEATS 1 and 2, austenitised at 1000°C, was determined as 181 and 227 carbides per mm<sup>2</sup>,

respectively. The carbides have an average diameter of 1.28  $\mu\text{m}$  in HEAT 1 and 0.75  $\mu\text{m}$  in HEAT 2. The measured hardness values are high at  $664\pm 12$  HV for HEAT 1 and  $639\pm 10$  HV for HEAT 2. These high hardness values can be attributed to the fine martensitic matrix and the low levels of retained austenite.

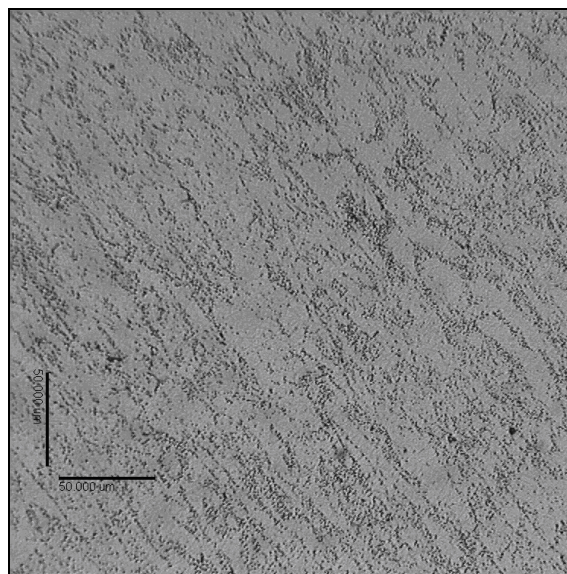


**Figure 4.4:** Scanning electron micrograph of HEAT 2 after austenitising for 15 minutes at  $1000^\circ\text{C}$ , followed by oil quenching. The microstructure consists of coarse  $\text{M}_{23}\text{C}_6$  carbides in a fine martensite matrix. (Hardness:  $639\pm 10$  HV).

According to Pickering<sup>2</sup>, the equilibrium carbide dissolution temperature in AISI 420 (without added molybdenum) is  $1050^\circ\text{C}$ . The heating rate used by Pickering was, however, not reported. According to De Andres *et al*<sup>16</sup> the total carbide dissolution temperature is, to a certain degree, dependent on the heating rate. A carbide dissolution temperature of  $1110^\circ\text{C}$  was reported for a heating rate of  $0.5^\circ\text{C}$  per second. Due to furnace constraints, a heating rate of approximately  $0.2^\circ\text{C}$  per second was used for the purpose of this investigation, and a relatively high percentage of carbides is therefore expected in the as-quenched microstructure after austenitising at  $1050^\circ\text{C}$ . Based on published literature, the carbide dissolution temperature, using a  $0.2^\circ\text{C}$  per second heating rate, is expected to be approximately  $1090^\circ\text{C}$ .

To determine the extent of carbide dissolution at  $1050^\circ\text{C}$ , samples from HEAT 1 and HEAT 2 were austenitised at  $1050^\circ\text{C}$  for 15 minutes, followed by quenching in oil. A representative optical photomicrograph of HEAT 1 in the as-quenched condition is

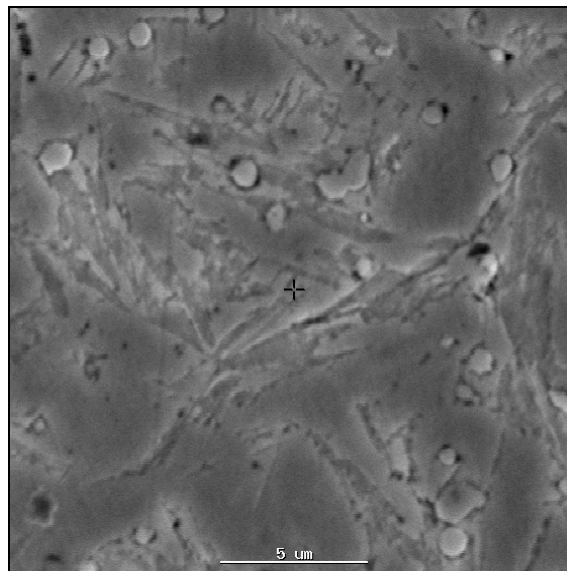
shown in Figure 4.5. The microstructure consists of well-defined carbide particles in a martensitic matrix. This confirms that an austenitising temperature of 1050°C is still below the temperature required to completely dissolve the  $M_{23}C_6$  precipitates in the two heats examined. Carbide densities of 131 and 184 carbides per  $mm^2$  were obtained for HEAT 1 and HEAT 2, respectively. These densities are lower than those observed in samples quenched from 1000°C, implying that partial dissolution of the carbides had occurred. The measured hardness values are  $678 \pm 9$  HV for HEAT 1 and  $665 \pm 9$  HV for HEAT 2. These hardness values are somewhat higher than those measured after quenching from 1000°C. This increase in hardness is attributed to an increase in the carbon content of the martensite phase due to the partial dissolution of carbides. The martensite is therefore harder due to its higher carbon content, but the amount of carbon and alloying elements in solid solution was not high enough to depress the martensite transformation range below 0°C. The retained austenite contents of the two heats therefore remain low at 5% and 6%.



**Figure 4.5:** Optical micrograph of HEAT 1 after austenitising for 15 minutes at 1050°C, followed by oil quenching. The microstructure consists of  $M_{23}C_6$  carbides in a fine martensite matrix. (Hardness:  $678 \pm 9$  HV). (Magnification: 200x).

Austenitising at a temperature of 1075°C yields as-quenched hardness values of  $684 \pm 10$  HV for HEAT 1 and  $674 \pm 12$  HV for HEAT 2. As shown in Figure 4.6, the microstructures of the two heats are predominantly martensitic, with well-defined carbide particles. Carbide densities of 87 and 117 carbides per  $mm^2$  were

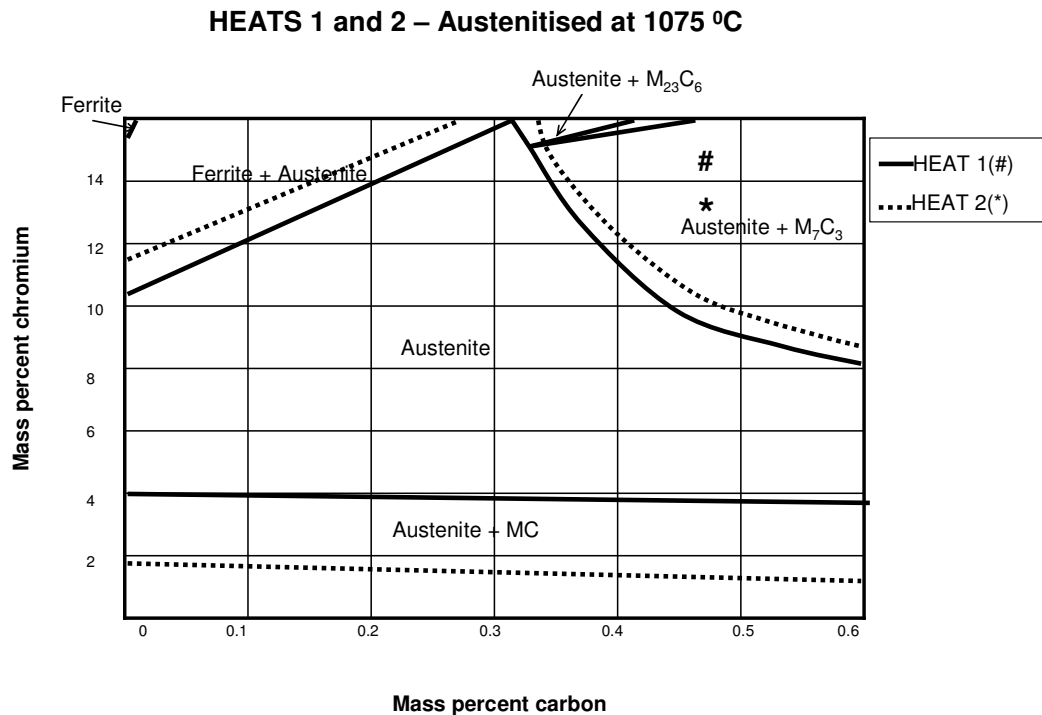
determined for HEATS 1 and 2, respectively. Although these densities are somewhat lower than those measured after austenitising at 1050°C, suggesting that partial dissolution of carbides had occurred, the results suggest that 1075°C is still below the complete carbide dissolution temperature. The average carbide particle diameters of 0.93 µm (HEAT 1) and 0.58 µm (HEAT 2) are very similar to those observed at lower austenitising temperatures. Partial dissolution of carbides increased the carbon and alloying element contents of the austenite, resulting in slightly higher as-quenched hardness values (compared to those recorded after quenching from 1050°C) and higher retained austenite levels of 15% and 10% in HEATS 1 and 2, respectively.



**Figure 4.6:** Scanning electron micrograph of HEAT 1 after austenitising for 15 minutes at 1075°C, followed by oil quenching. The microstructure consists of  $M_{23}C_6$  carbides in a martensite matrix. (Hardness:  $684 \pm 10$  HV).

Published literature<sup>1,6,16</sup> suggests that extensive carbide dissolution should occur in both heats during austenitising at 1075°C. In order to explain the observed discrepancy between published dissolution temperatures and the microstructures observed after austenitising at 1075°C, thermodynamic predictions (using the CALPHAD™ model) of the austenite and carbide stabilities in the two heats during austenitising were used. Figure 4.7 shows the predicted equilibrium phase diagram for HEATS 1 and 2 at 1075 °C. In this figure, the # symbol denotes the position of HEAT 1 in terms of percentage chromium and percentage carbon, whereas \*

denotes the position of HEAT 2. The solid lines represent the calculated phase boundaries for HEAT 1 and the broken lines the boundaries for HEAT 2. It is evident from Figure 4.7 that the higher molybdenum and chromium contents of HEAT 1 restrict the austenite phase field (both elements are strong ferrite-formers).

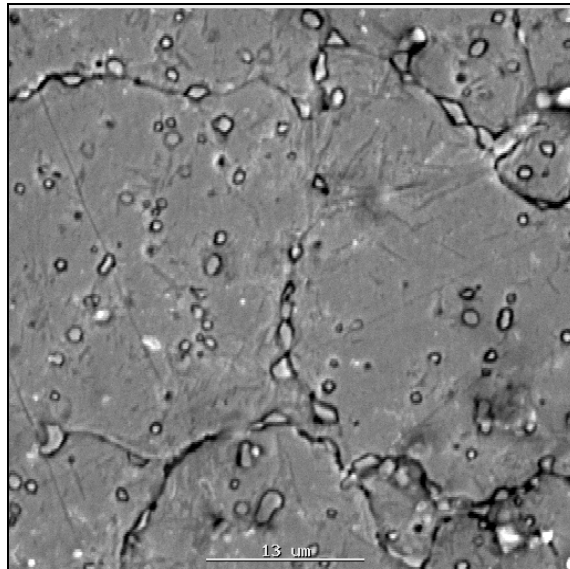


**Figure 4.7:** Thermodynamic prediction of the equilibrium phase diagram of two heats of AISI 420 at an austenitising temperature of 1075 °C.

Figure 4.7 predicts that both heats contain austenite and  $M_7C_3$  carbides during austenitising at 1075 °C. According to the predicted phase diagram all  $M_{23}C_6$  carbides are in solution at this temperature. This is in agreement with the observations of Salem<sup>24</sup>, who reported that  $M_{23}C_6$  chromium carbides dissolve in the 950 °C to 1050 °C temperature range, whereas  $M_7C_3$  carbides dissolve in the 1050 °C to 1150 °C temperature range. Partial dissolution of  $M_7C_3$  carbides is therefore likely at 1075 °C. Information derived from the CALPHAD model predicts 1.23%  $M_7C_3$  in HEAT 1 at 1075 °C, and 0.91%  $M_7C_3$  in HEAT 2. No  $M_{23}C_6$  is expected in these steels at 1075 °C, but  $M_{23}C_6$  forms during cooling on conversion of the  $M_7C_3$  carbides. The presence of  $M_{23}C_6$  carbide in the as-quenched microstructures of the steels was confirmed by XRD analysis. No  $M_7C_3$  was observed in any of the steels after cooling from the austenitising temperature.



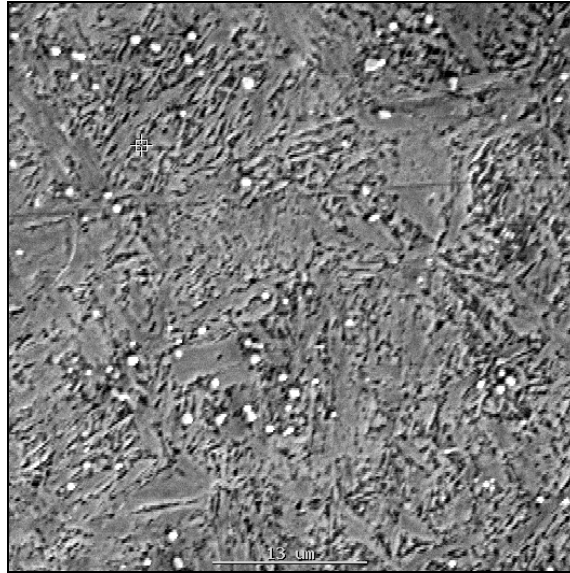
Available literature predicts that increasing the austenitising temperature to 1100°C should result in a large percentage of carbides going into solution. The critical temperature for complete carbide solution in AISI 420 (without molybdenum) was recorded as 1110°C at a heating rate of 0.5°C per second<sup>16</sup>. At the 0.2°C per second heating rate used in the current investigation, a carbide dissolution temperature of approximately 1090°C is predicted. Figures 4.8 and 4.9 indicate, however, that carbides remain present in the as-quenched microstructures of both HEAT 1 and HEAT 2. The carbide densities were estimated as 84 carbides per mm<sup>2</sup> for HEAT 1 and 63 carbides per mm<sup>2</sup> for HEAT 2. XRD analysis reported 23% retained austenite in HEAT 1, and 12% retained austenite in HEAT 2. The increase in retained austenite content accounts for the lower as-quenched hardness values of 653±8 HV for HEAT 1 and 639±8 HV for HEAT 2.



**Figure 4.8:** Scanning electron micrograph of HEAT 1 after austenitising for 15 minutes at 1100°C, followed by oil quenching. The microstructure consists of  $M_{23}C_6$  carbides in a martensite matrix containing 23% retained austenite. (Hardness: 653±8 HV).

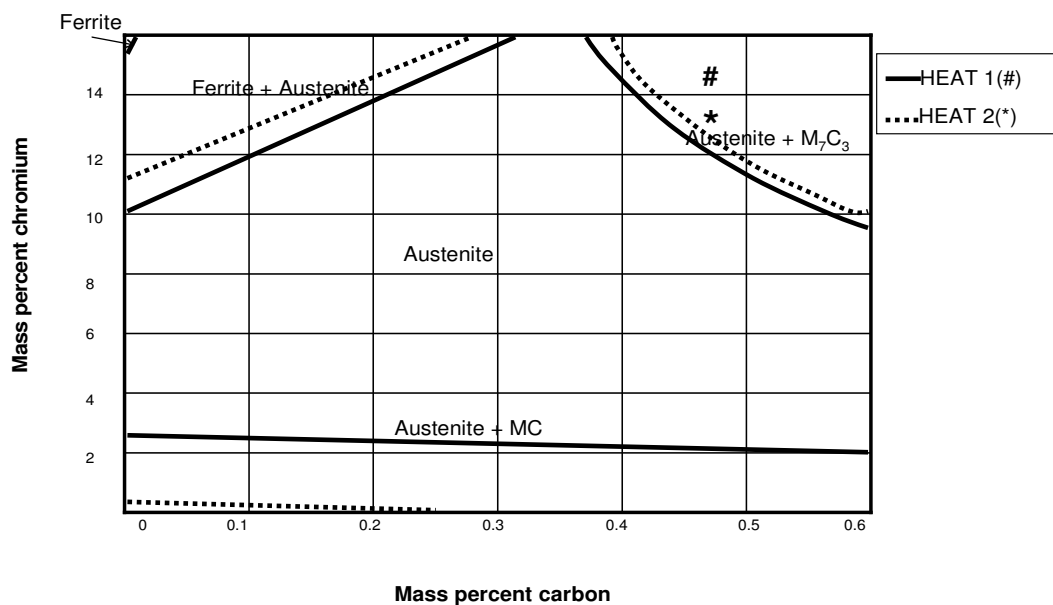
This inconsistency between the reported carbide dissolution temperatures and those determined during the course of this investigation was investigated further by examining the predicted phase diagram at 1100°C (shown in Figure 4.10). At this temperature, both steels are located in the dual-phase region where austenite and  $M_7C_3$  carbides are stable, suggesting that the temperature is not high enough to completely dissolve all carbides. Extensive carbide dissolution does, however, take place at this temperature, with the CALPHAD model predicting 0.62%  $M_7C_3$  in HEAT

1 and 0.27% in HEAT 2 at 1100 °C. The increased dissolution of carbides depresses the martensite transformation range, resulting in the formation of higher levels of retained austenite.



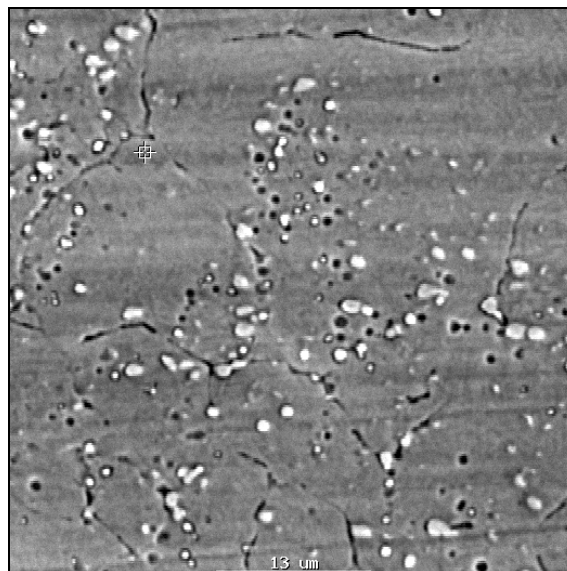
**Figure 4.9:** Scanning electron micrograph of HEAT 2 after austenitising for 15 minutes at 1100 °C, followed by oil quenching. The microstructure consists of  $M_{23}C_6$  carbides in a martensite matrix containing 12% retained austenite. (Hardness: 639±15 HV).

#### HEATS 1 and 2 – Austenitised at 1100 °C



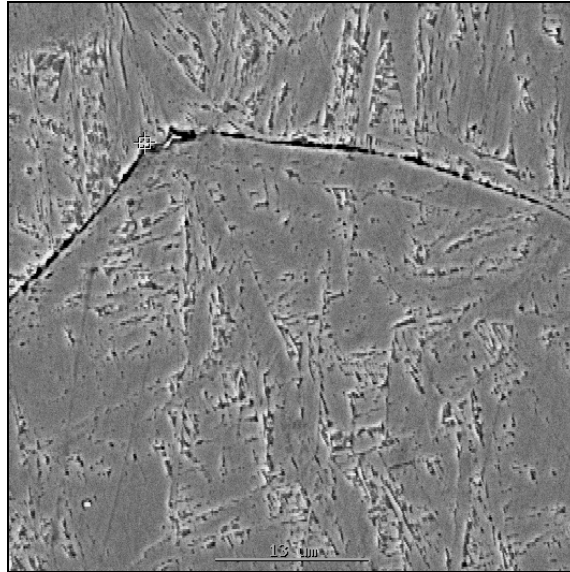
**Figure 4.10:** Thermodynamic prediction of the equilibrium phase diagram of two heats of AISI 420 at an austenitising temperature of 1100 °C.

Scanning electron micrographs of HEATS 1 and 2 after austenitising at 1130°C are shown in Figures 4.11 and 4.12. Contrary to published predictions, carbides remain visible in the as-quenched microstructures, with measured carbide densities of 81 carbides per mm<sup>2</sup> for HEAT 1 and 32 carbides per mm<sup>2</sup> for HEAT 2. The hardness of HEAT 1 is, however, significantly lower than that of HEAT 2 at 474±7 HV. This can be attributed to the presence of a significant volume fraction of retained austenite (25%) after austenitising at 1130°C. The hardness of HEAT 2 remains high at 620±4 HV, which is in agreement with the measured retained austenite content of 15%. A significant increase in ASTM grain size is observed at this austenitising temperature, with ASTM grain size numbers of 8.6 for HEAT 1 and 6.9 for HEAT 2. This increase in grain size can be attributed to the higher austenitising temperature and the increased dissolution of grain pinning carbides.



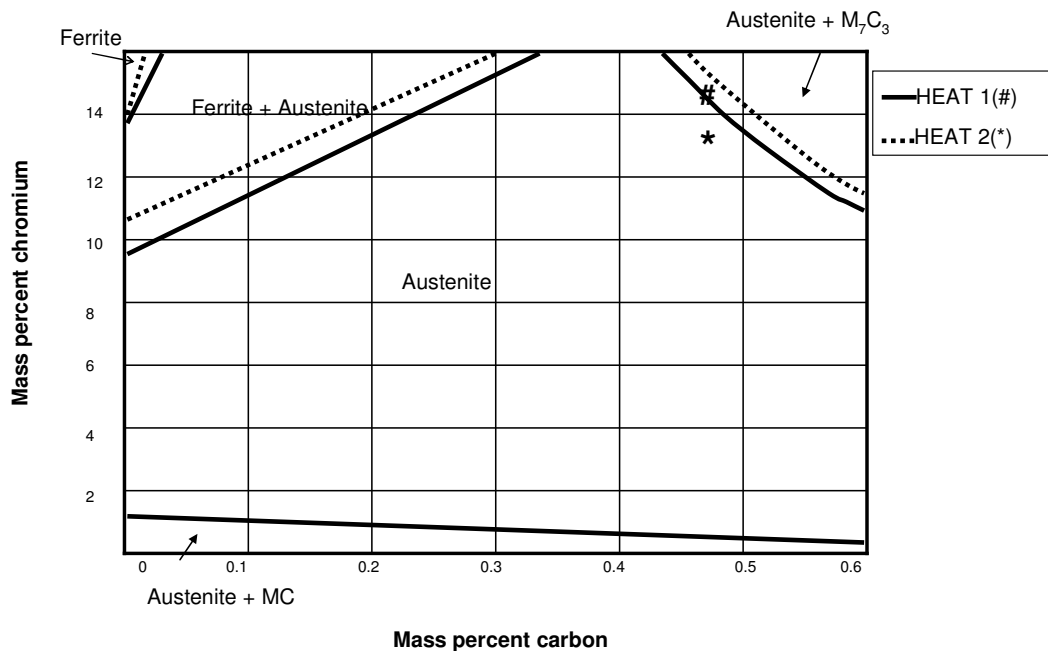
**Figure 4.11:** Scanning electron micrograph of HEAT 1 after austenitising for 15 minutes at 1130°C, followed by oil quenching. The microstructure consists of  $M_{23}C_6$  carbides in a martensite matrix containing 25% retained austenite. (Hardness: 474±7 HV).

The predicted phase diagrams for HEATS 1 and 2 at 1130°C are shown in Figure 4.13. HEAT 1 is located on the boundary between the austenite and (austenite +  $M_7C_3$ ) phase fields, whereas HEAT 2 is located well within the single-phase austenite region. The CALPHAD model therefore predicts complete dissolution of carbides during austenitising at 1130 °C. This suggests that equilibrium was not reached during heat treatment, resulting in the presence of retained carbides, or that the model does not predict the phase boundaries to the desired level of accuracy.



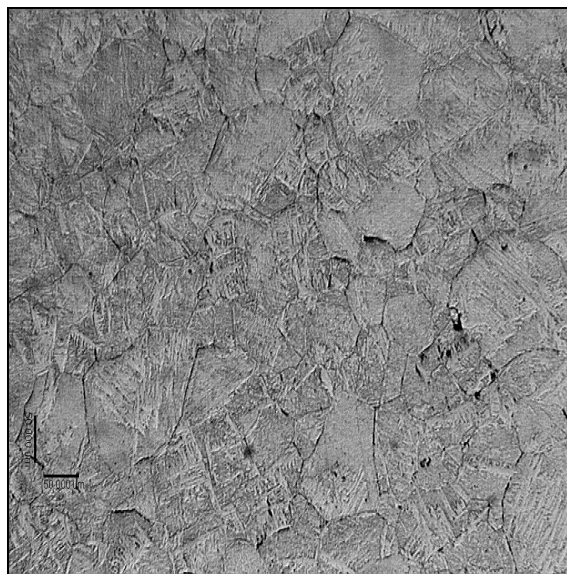
**Figure 4.12:** Scanning electron micrograph of HEAT 2 after austenitising for 15 minutes at 1130 °C, followed by oil quenching. The microstructure consists of  $M_{23}C_6$  carbides in a martensite matrix containing 15% retained austenite. (Hardness:  $620 \pm 4$  HV).

### HEATS 1 and 2 – Austenitised at 1130 °C



**Figure 4.13:** Thermodynamic prediction of the equilibrium phase diagram of two heats of AISI 420 at an austenitising temperature of 1130 °C.

Austenitising at a temperature of 1150°C results in a significant increase in retained austenite after quenching. HEAT 1 contains 27% retained austenite (see Figure 4.14), resulting in a low as-quenched hardness of  $308\pm 6$  HV. HEAT 2 contains approximately 17% retained austenite and displays a higher hardness of  $609\pm 10$  HV. The carbides have almost completely gone into solution, with residual carbide densities of 43 and 14 carbides per  $\text{mm}^2$  for HEATS 1 and 2, respectively. Due to the higher austenitising temperature and the dissolution of grain pinning carbides, considerable grain growth is observed and the average ASTM grain size number decreases to 6.3 for HEAT 1 and 5.1 for HEAT 2.

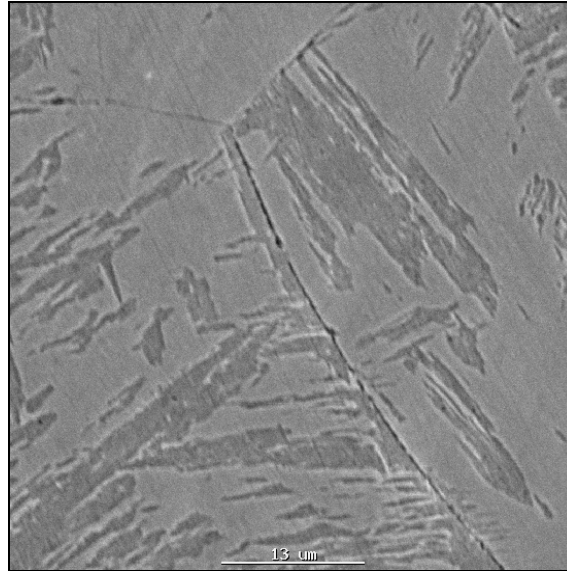


**Figure 4.14:** Optical micrograph of HEAT 1 after austenitising for 15 minutes at 1150°C, followed by oil quenching. The microstructure consists of a small number of residual  $M_{23}C_6$  carbides in a martensite matrix containing 27% retained austenite. (Hardness:  $308\pm 6$  HV). (Magnification: 100x).

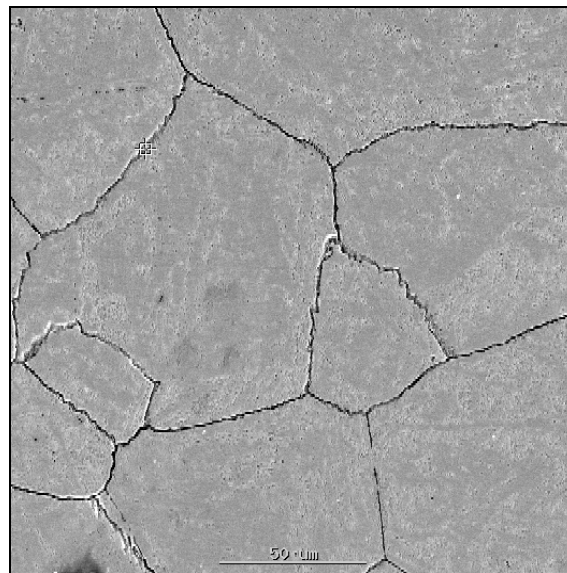
No phase diagram was calculated for an austenitising temperature of  $1150^\circ\text{C}$ , but both heats are expected to be located well within the austenite phase field at this temperature.

Scanning electron micrographs of HEATS 1 and 2 after austenitising at 1175°C are shown in Figures 4.15 and 4.16. Both heats contain martensite and retained austenite (29% retained austenite in HEAT 1 and 21% in HEAT 2). No carbide particles are visible, suggesting that this temperature is above the temperature for complete carbide dissolution in both alloys. Excessive grain growth is evident in both

heats, with average ASTM grain size numbers of 4 and 3.4 for HEATS 1 and 2. The high percentage retained austenite resulted in low hardness values of  $279\pm 4$  HV for HEAT 1 and  $488\pm 3$  HV for HEAT 2.

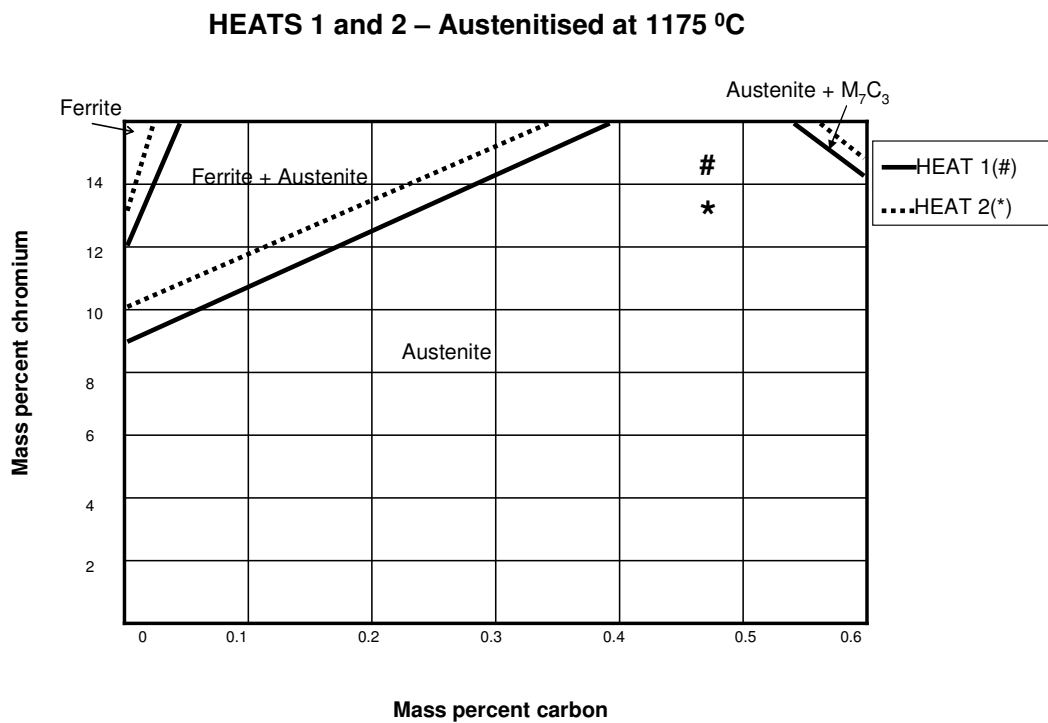


**Figure 4.15:** Scanning electron micrograph of HEAT 1 after austenitising for 15 minutes at  $1175^{\circ}\text{C}$ , followed by oil quenching. The microstructure consists of martensite and 29% retained austenite. (Hardness:  $279\pm 4$  HV).



**Figure 4.16:** Scanning electron micrograph of HEAT 2 after austenitising for 15 minutes at  $1175^{\circ}\text{C}$ , followed by oil quenching. The microstructure consists of martensite and 21% retained austenite. (Hardness:  $488\pm 3$  HV).

The CALPHAD model predicts that no carbides are present in either heat at an austenitising temperature of 1175°C. As shown in Figure 4.17, both alloys are located well within the austenite phase field. The complete dissolution of carbides during heat treatment depresses the martensite transformation range and results in high levels of retained austenite.

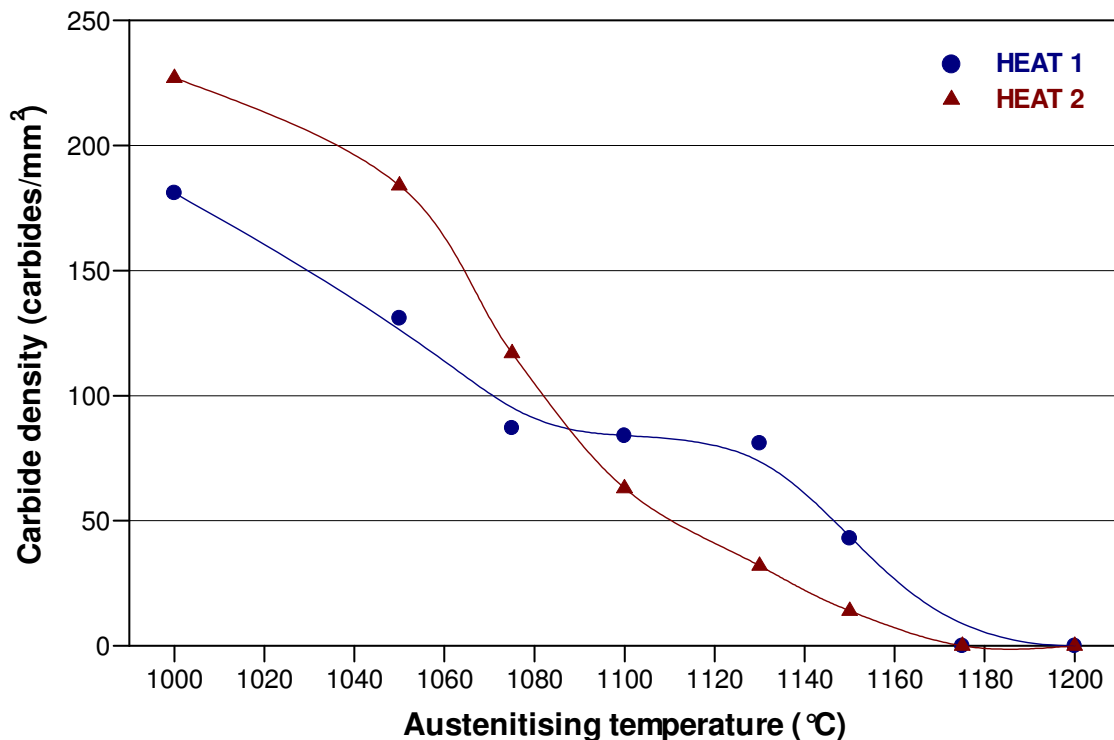


**Figure 4.17:** Thermodynamic prediction of the equilibrium phase diagram of two heats of AISI 420 at an austenitising temperature of 1175°C.

At 1200°C, all carbides are in solution in the austenite, resulting in as-quenched microstructures containing martensite and retained austenite. HEAT 1 contains 33% retained austenite, whereas HEAT 2 contains 24%. Hardness values of  $270 \pm 12$  HV for HEAT 1 and  $459 \pm 2$  HV for HEAT 2 were measured. The higher hardness of HEAT 2 can be attributed to the higher martensite content after quenching. Excessive grain growth occurs at the austenitising temperature, yielding average as-quenched ASTM grain size numbers of 2.8 and 3.2 for HEATS 1 and 2, respectively.

A brief summary of these results is given below.

Table 4.1 confirms that an increase in austenitising temperature is associated with a decrease in carbide density and an increase in the percentage retained austenite. Figure 4.18 shows the effect of austenitising temperature on the carbide density in HEATS 1 and 2. The carbide densities in both steels decrease with an increase in austenitising temperature. At austenitising temperatures between 1000°C and 1050°C, the carbide densities of both heats decrease at similar rates, suggesting the progressive dissolution of  $M_{23}C_6$  carbides ( $M_{23}C_6$  is reported to dissolve at temperatures between 950°C and 1050°C). HEAT 2 has a higher initial carbide density, but at temperatures higher than 1050°C the carbide density decreases at a faster rate than that of HEAT 1. This suggests that the increased molybdenum content of HEAT 1 retards carbide dissolution at higher austenitising temperatures, possibly due to the higher stability of  $M_7C_3$  in the molybdenum-containing alloy (as confirmed by phase diagrams constructed using the CALPHAD model). Molybdenum appears to stabilise and promote the equilibrium  $M_7C_3$  carbide at temperatures higher than 1050°C, retarding carbide coarsening and dissolution. Complete carbide dissolution takes place at temperatures higher than 1175°C in both heats.

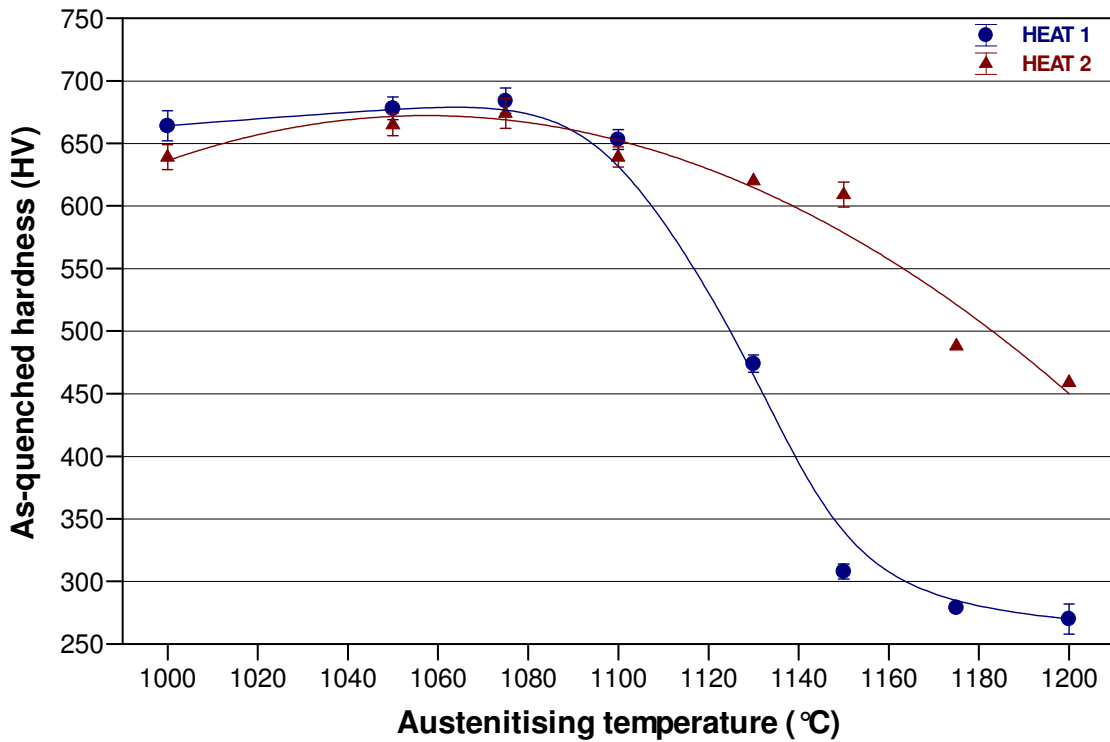


**Figure 4.18:** The influence of austenitising temperature on the carbide density (number of carbides per mm<sup>2</sup>) in HEATS 1 and 2.

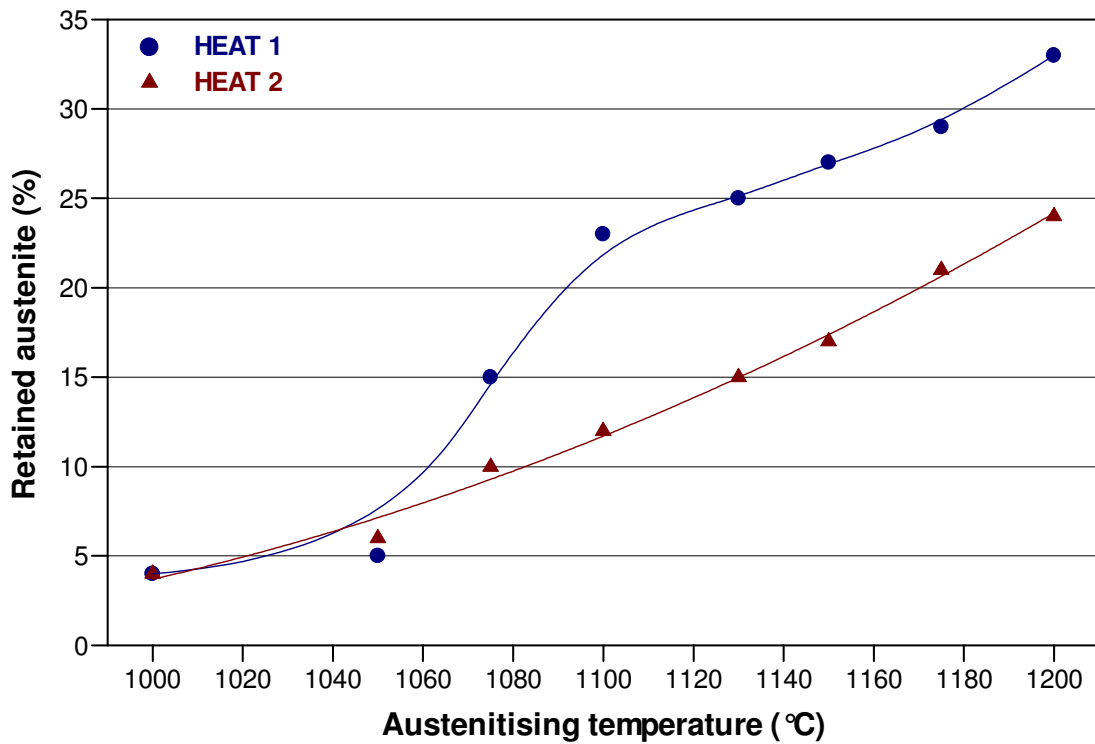


The gradual dissolution of carbides up to austenitising temperatures of 1175°C (where complete carbide dissolution is observed in both heats) affects the measured retained austenite content, as-quenched hardness and grain size of the steels. The effect of austenitising temperature on the as-quenched hardness is shown in Figure 4.19. The measured hardness values of both heats increase slightly with an increase in austenitising temperature up to 1075°C. This can be attributed to the gradual dissolution of  $M_{23}C_6$  carbides, which raises the carbon content of the austenite phase at elevated temperatures. On quenching a higher carbon martensite forms, which increases the as-quenched hardness of both heats. Although retained austenite is present in both heats after quenching from austenitising temperatures of 1075°C or lower, retained austenite is not present in high enough quantities to reduce the as-quenched hardness significantly. Higher austenitising temperatures raise the amount of carbon and alloying elements in solution in the austenite, and depress the martensite transformation range to lower temperatures. At temperatures higher than approximately 1075°C, increased carbide dissolution results in higher retained austenite contents (as shown in Figure 4.20), particularly in HEAT 1, and a considerable reduction in hardness. The higher retained austenite content and lower as-quenched hardness of HEAT 1 after austenitising at higher temperatures can probably be attributed to the higher alloying content of this steel. More molybdenum in solid solution causes a considerable reduction in the martensite transformation range, resulting in higher levels of retained austenite.

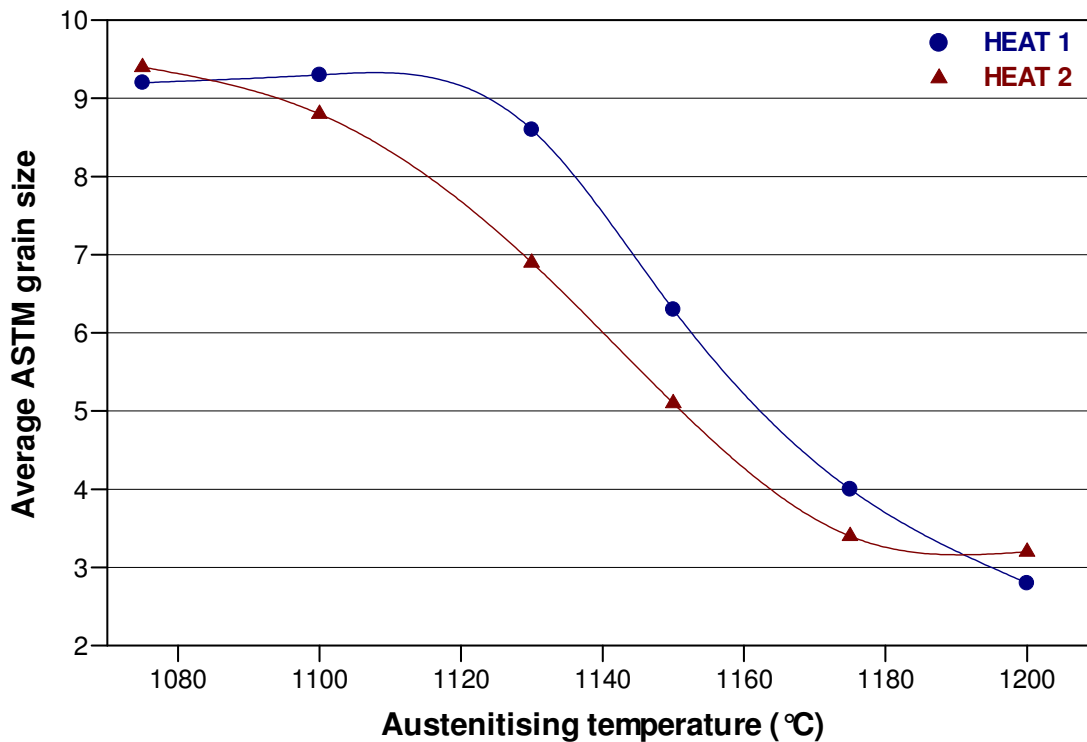
The dissolution of carbides during austenitising also affects the austenite grain size, as shown in Figure 4.21 for austenitising temperatures between 1000°C and 1100°C. The average ASTM grain size number remains stable at well above 9 for austenitising temperatures below about 1075°C. At austenitising temperatures between 1075°C and 1200°C, the grain size increases rapidly from an average ASTM grain size number of 9 to around 3. This increase in grain size is associated with the increase in temperature (providing a higher driving force for grain growth during heat treatment), compounded by coarsening and dissolution of grain pinning carbides. Grain growth in HEAT 1 is suppressed at temperatures below approximately 1120°C due to the higher stability of carbides in this steel. Once all the alloying elements are in solid solution due to the dissolution of carbides at higher temperatures, the austenite grain sizes of the two heats are similar.



**Figure 4.19:** The influence of austenitising temperature on the as-quenched hardness of HEATS 1 and 2 (with 95% confidence interval).



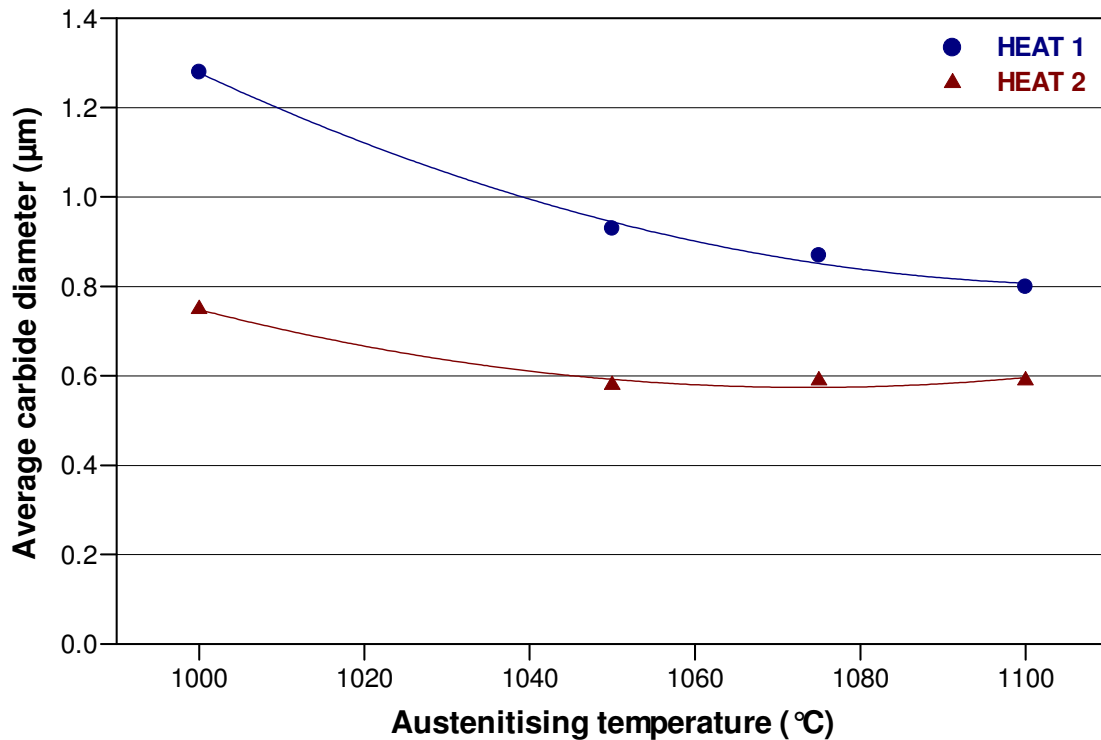
**Figure 4.20:** The influence of austenitising temperature on the as-quenched retained austenite content of HEATS 1 and 2.



**Figure 4.21:** The influence of austenitising temperature on the average ASTM grain size of HEATS 1 and 2.

The average carbide diameters of the two heats are shown in Figure 4.22 as a function of austenitising temperature. The average carbide diameter measured in HEAT 1 decreases from approximately 1.3  $\mu\text{m}$  at 1000°C to 0.8  $\mu\text{m}$  at 1100°C. This reduction in average carbide diameter is less evident in HEAT 2, with the diameter decreasing from approximately 0.75  $\mu\text{m}$  at 1000°C to below 0.6  $\mu\text{m}$  diameter at 1100°C.

It is evident from Table 4.1 and Figure 4.19 that peak hardness values of  $684 \pm 10$  HV and  $674 \pm 12$  HV are obtained in HEAT 1 and HEAT 2, respectively, after quenching from an austenitising temperature of 1075°C. Although these hardness values are well above the minimum requirement of 610 HV stated in Chapter 2, the steels contain between 10 and 15% retained austenite after quenching. Since minimal retained austenite is specified as a requirement, the effect of tempering on the microstructure (and in particular the retained austenite content) and the properties of the two heats was investigated.



**Figure 4.22:** The influence of austenitising temperature on the average carbide diameter in HEATS 1 and 2.

### **3. THE EFFECT OF TEMPERING ON THE MICROSTRUCTURE AND PROPERTIES OF HEATS 1 AND 2**

As stated in Chapter 2, a martensitic structure with minimal retained austenite, an evenly dispersed carbide structure and hardness values of between 610 HV and 740 HV are required to satisfy the design criteria for the steels examined during the course of this investigation. Austenitising at 1075°C for 15 minutes, followed by oil quenching, yields martensitic structures with dispersed carbides and hardness values well within the specified range for HEATS 1 and 2. The retained austenite contents of both steels are, however, higher than the desired levels.

To increase the toughness of the as-quenched martensitic structures, transform retained austenite and induce secondary hardening, both heats of medium-carbon AISI 420 martensitic stainless steel were tempered at three temper temperatures, namely 550°C, 650°C and 750°C, for 30 minutes. Since tempering reduces the as-quenched hardness of both heats to unacceptable levels after austenitising at temperatures below 1150°C, the following discussion considers only the results obtained on tempering HEATS 1 and 2 after austenitising at 1150°C and 1175°C.

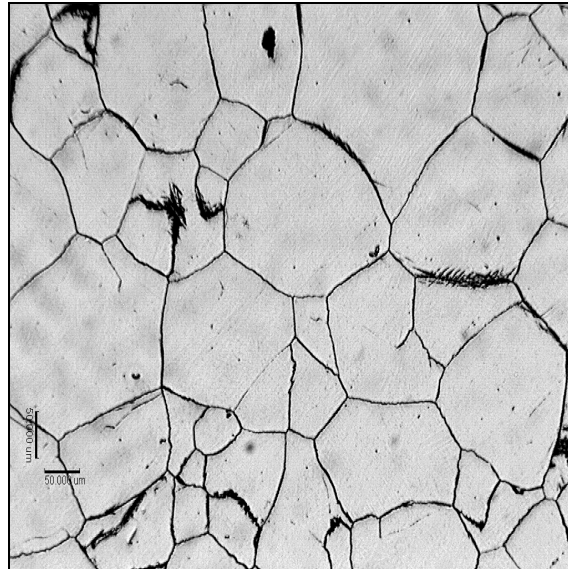
### **3.1 Tempering after austenitising at 1150°C or 1175°C:**

The hardness of HEAT 1 after quenching from 1150°C was  $308\pm 6$  HV. Tempering at 550°C has very little effect on the microstructure of this alloy. Retained austenite and martensite was observed in the tempered microstructure, and an average hardness of  $299\pm 10$  HV was recorded. Increasing the tempering temperature to 650°C increased the hardness to  $362\pm 14$  HV, whereas tempering at 750°C raised the hardness to  $560\pm 10$  HV. This increase in hardness can be attributed to the formation of secondary alloy carbides, and most likely the transformation of some retained austenite to martensite. These hardness values are, however, still well below the required hardness level. The as-quenched hardness of HEAT 2 after austenitising at 1150°C for 15 minutes was  $609\pm 10$  HV. Tempering at 550°C yielded a microstructure containing residual austenite and martensite, with a hardness of  $513\pm 9$  HV. Tempering at 650°C reduced the hardness to  $482\pm 14$  HV, while tempering at 750°C reduced the hardness even further to  $400\pm 10$  HV. This steel has a lower alloying element content (lower molybdenum level) and therefore does not respond to secondary hardening to the same extent as the molybdenum-alloyed HEAT 1. None of these treatments satisfy the requirements stated in Chapter 2.

Austenitising at 1175°C, followed by oil quenching, yielded hardness values of  $279\pm 4$  HV for HEAT 1 and  $488\pm 3$  HV for HEAT 2. Subsequent tempering of HEAT 1 at 550°C has very little effect on the microstructure of the sample (as shown in Figure 4.23), and an average hardness of  $284\pm 14$  HV was recorded. Raising the tempering temperature to 650°C raised the hardness to  $409\pm 13$  HV due to alloy carbide precipitation and partial transformation of retained austenite to martensite. Very little change in microstructure or hardness was observed for HEAT 2 after tempering at 550°C or 650°C. Due to the low hardness values recorded, tempering at 750°C was not performed.

The measured hardness values of HEATS 1 and 2 after austenitising at 1150°C and 1175°C, followed by tempering at 550°C, 650°C or 750°C, are compared in Table 4.2. The hardness values of HEAT 1 exhibit a characteristic increase during tempering. This suggests that the higher molybdenum content of HEAT 1 promoted secondary hardening in this alloy. The precipitation of fine alloy carbides during tempering is also expected to increase the martensite transformation range and

facilitate the partial transformation of retained austenite to martensite. The hardness of HEAT 2 decreased during tempering. The absence of molybdenum and the lower chromium content of this steel reduced the temper resistance of steel and limited the extent of secondary hardening.



**Figure 4.23:** Optical micrograph of HEAT 1 after austenitising at 1175°C, followed by oil quenching and tempering at 550°C for 30 minutes. The microstructure consists of martensite and retained austenite. (Hardness: 284±14 HV). (Magnification: 100x).

**Table 4.2:** The effect of tempering on the hardness of HEATS 1 and 2 after austenitising at 1150°C and 1175°C.

Austenitising temperature	HEAT 1			HEAT 2		
	As-quenched hardness	Temper temperature	Hardness	As-quenched hardness	Temper temperature	Hardness
1150°C	308±6 HV	550°C	299±10 HV	609±10 HV	550°C	513±9 HV
		650°C	362±14 HV		650°C	482±14 HV
		750°C	560±10 HV		750°C	400±10 HV
1175°C	279±4 HV	550°C	284±14 HV	488±3 HV	550°C	446±5 HV
		650°C	409±13 HV		650°C	455±8 HV

### 3.2 Double tempering:

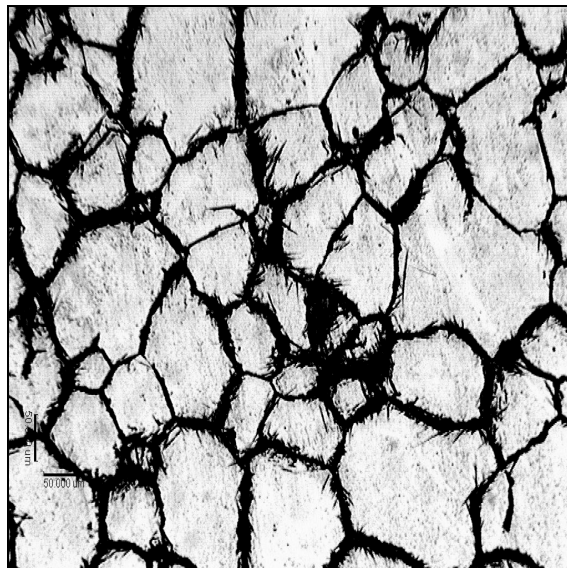
In order to promote the transformation of retained austenite to martensite, a second tempering step was introduced for samples austenitised at 1150°C and tempered at 650°C or 750°C, and for samples austenitised at 1175°C and tempered at 550°C or 650°C. These samples were tempered for a second time at the same temper

temperatures (for an additional 30 minutes). The results obtained after double tempering are summarised in Table 4.3.

**Table 4.3:** The effect of double tempering on the hardness of HEATS 1 and 2 after austenitising at 1150°C and 1175°C.

Austenitising temperature	HEAT 1			HEAT 2		
	Temper temperature	Hardness (1 <sup>st</sup> temper)	Hardness (2 <sup>nd</sup> temper)	Temper temperature	Hardness (1 <sup>st</sup> temper)	Hardness (2 <sup>nd</sup> temper)
1150°C	650°C	362±14 HV	500±9 HV	650°C	482±14 HV	360±5 HV
	750°C	560±10 HV	320±7 HV	750°C	400±10 HV	294±7 HV
1175°C	550°C	284±14 HV	295±14 HV	550°C	446±5 HV	522±5 HV
	650°C	409±13 HV	388±9 HV	650°C	455±8 HV	344±6 HV

With the exception of HEAT 1 austenitised at 1150°C and double tempered at 650°C, and HEAT 2 austenitised at 1175°C and double tempered at 550°C, double tempering did not result in any significant hardening in any of the samples tested. An optical photomicrograph of HEAT 1 after austenitising at 1150°C and double tempering at 650°C is shown in Figure 4.24. Even though double tempering results in a hardness increase in this sample, the microstructure still contains a significant amount of retained austenite after the double temper treatment. All hardness values in Table 4.3 are well below the required hardness values.



**Figure 4.24:** Optical micrograph of HEAT 1 after austenitising at 1150°C, followed by oil quenching and double tempering at 650°C. The microstructure consists of martensite and retained austenite. (Hardness: 500±9 HV). (Magnification: 100x).

It is apparent from the preceding discussion that the microstructure and properties obtained after tempering AISI 420 at temperatures between 550°C and 750°C after austenitising at 1150°C or 1175°C do not satisfy the requirements stated in Chapter 2. In order to facilitate a higher degree of transformation of retained austenite to martensite, cryogenic treatment was performed. The results of this investigation are described below.

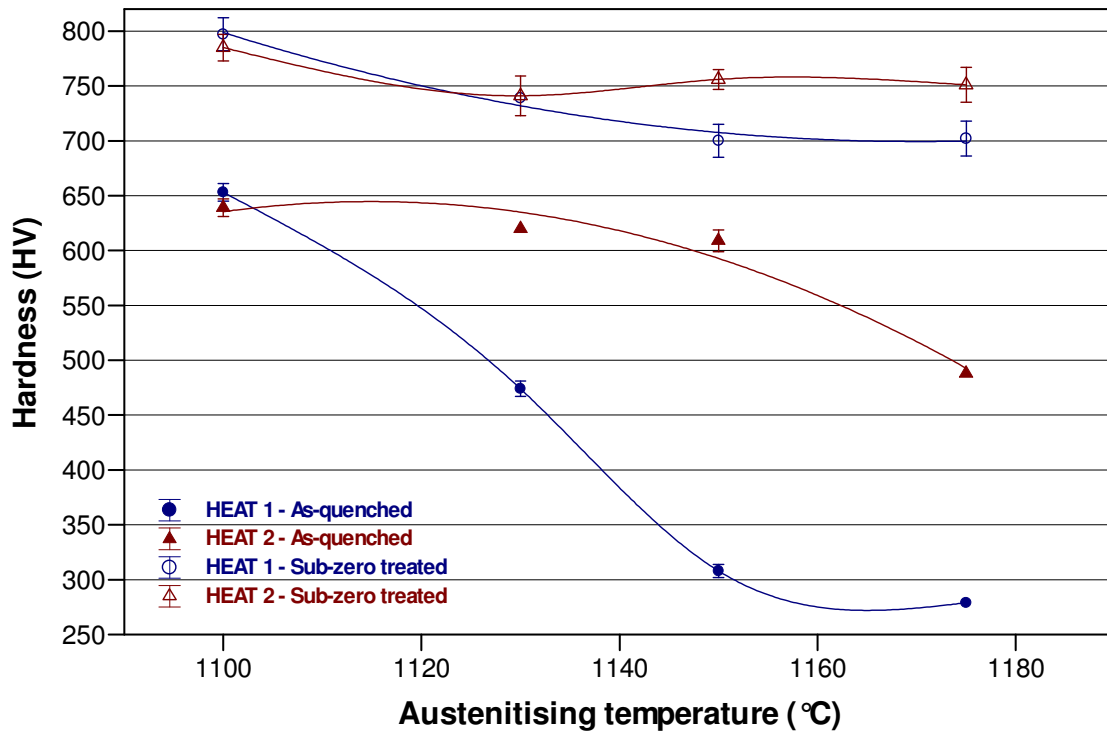
### **3.3 Sub-zero treatments:**

In order to determine the effect of sub-zero tempering on the microstructure and hardness of HEATS 1 and 2, samples austenitised at 1100°C, 1130°C, 1150°C and 1175°C were sub-zero tempered in liquid nitrogen at -196°C for 40 minutes after oil quenching.

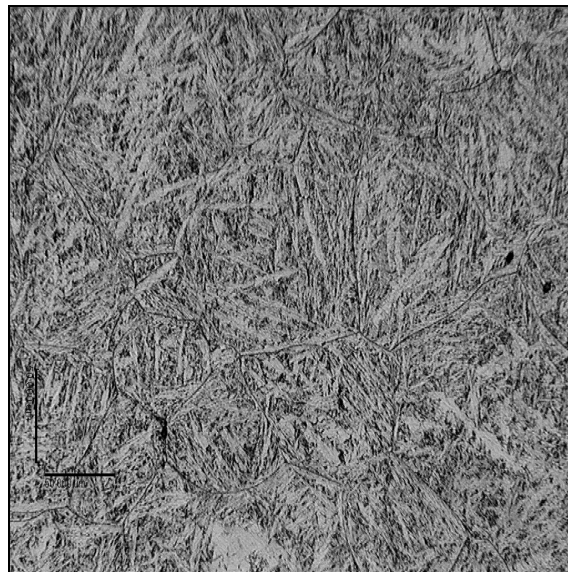
The influence of sub-zero treatment after quenching is illustrated in Figure 4.25 as a function of austenitising temperature for HEATS 1 and 2. Treatment in liquid nitrogen raised the hardness values considerably in both HEAT 1 and HEAT 2, with final hardness values above 690 HV in all the samples evaluated. This suggests that the majority of the retained austenite in the as-quenched microstructures transformed to martensite during sub-zero treatment, resulting in a significant increase in hardness. All samples of HEAT 1 displayed hardness values higher than 700 HV, a substantial increase over those measured in the as-quenched condition (particularly at higher austenitising temperatures). The recorded hardness values of HEAT 2 ranged between 739 HV and 785 HV after sub-zero tempering, as opposed to 639 HV to 488 HV for the as-quenched samples. A representative example of the microstructure of HEAT 2 after sub-zero treatment is shown in Figure 4.26. The structure is predominantly martensitic, with a small percentage of retained austenite.

The results shown in Figure 4.25 suggest that sub-zero treatment in liquid nitrogen is effective in transforming most of the retained austenite to martensite. The final hardness values are, however, too high to satisfy the design requirements of the steel, and the newly transformed martensite most likely too brittle. HEATS 1 and 2 were therefore tempered at 550°C or 700°C after austenitising at 1130°C, 1150°C and 1175°C, oil quenching and sub-zero treatment at -196°C. A tempering time of 1 hour was used. The results of tempering after sub-zero treatment in liquid nitrogen are considered below.



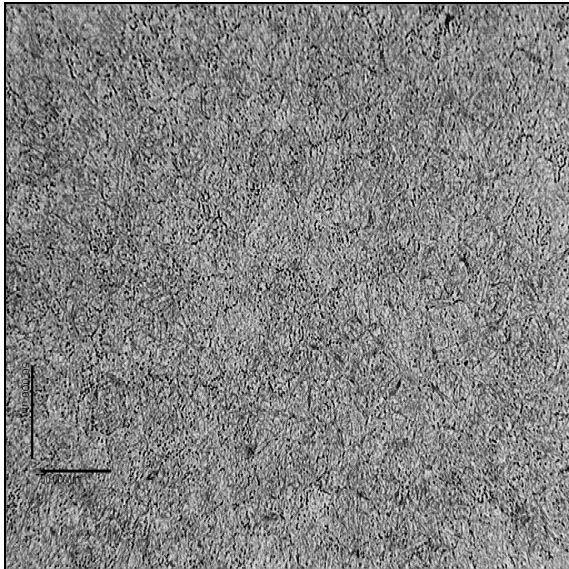


**Figure 4.25:** The effect of sub-zero tempering on the hardness of HEATS 1 and 2 after austenitising at temperatures between 1100°C and 1175°C.

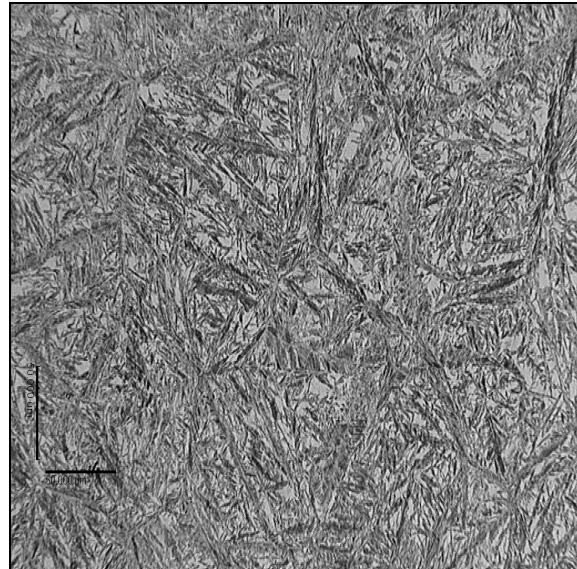


**Figure 4.26:** Optical micrograph of HEAT 2 after austenitising at 1130°C, followed by oil quenching and sub-zero treatment in liquid nitrogen. The microstructure consists of martensite and a small amount of retained austenite. (Hardness:  $741 \pm 18$  HV). (Magnification: 200x).

HEAT 1, austenitised at 1130 °C to 1175 °C, oil quenched, sub-zero treated at -196 °C and tempered at 550 °C, has final hardness values between 649 HV and 673 HV. Micrographs of HEAT 1, austenitised at 1130 °C, 1150 °C or 1175 °C, sub-zero treated at -196 °C and tempered at 550 °C, are shown in Figures 4.27, 4.28 and 4.29.

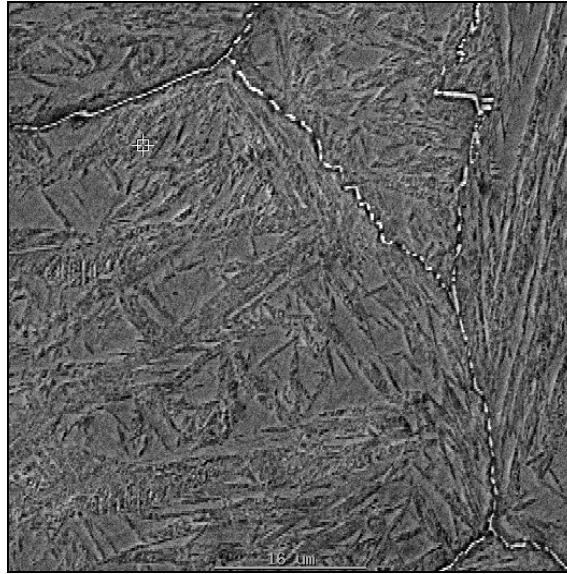


**Figure 4.27:** Optical micrograph of HEAT 1 after austenitising at 1130 °C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 550 °C. The microstructure consists of martensite and 3% retained austenite. (Hardness: 673±6 HV). (Magnification: 200x).



**Figure 4.28:** Optical micrograph of HEAT 1 after austenitising at 1150 °C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 550 °C. The microstructure consists of martensite and 5% retained austenite. (Hardness: 651±14 HV). (Magnification: 200x).

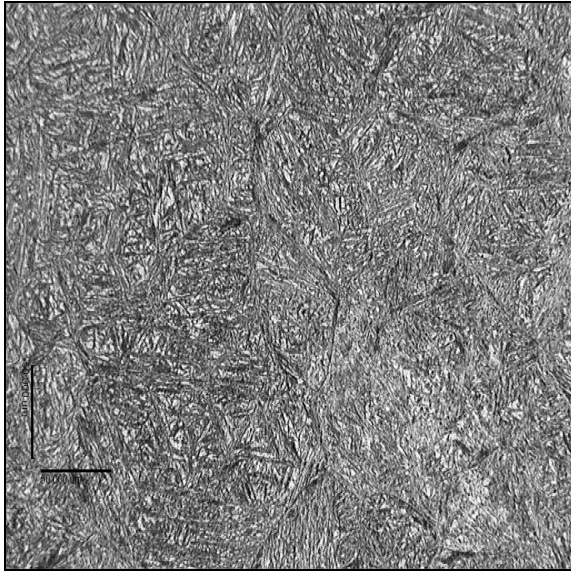
The microstructures shown in Figures 4.27 and 4.28 (for austenitising temperatures of 1130 °C and 1150 °C, respectively) consist of martensite, retained austenite and a small number of carbides. Retained austenite levels of 3% (austenitised at 1130 °C) and 5% (austenitised at 1150 °C) were measured. The microstructure of HEAT 1, austenitised at 1175 °C and subjected to sub-zero treatment and tempering at 550 °C, is shown in Figure 4.29. This microstructure consists of martensite, approximately 8% retained austenite and grain boundary  $M_{23}C_6$  carbide particles. The grain boundary  $M_{23}C_6$  carbides precipitate during tempering from the carbon saturated matrix. Since the requirements stated in Chapter 2 specify an evenly dispersed carbide network, this sample is not considered acceptable in view of the alloy design criteria.



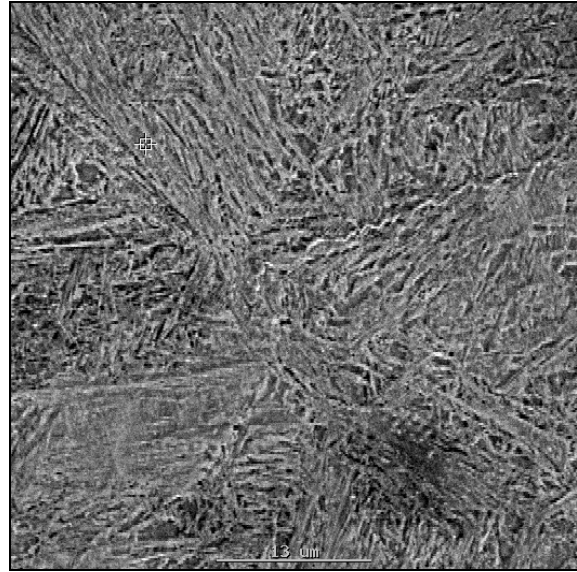
**Figure 4.29:** Scanning electron micrograph of HEAT 1 after austenitising at 1175°C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 550°C. The microstructure consists of martensite, 8% retained austenite and networks of intergranular  $M_{23}C_6$  carbides. (Hardness:  $649\pm 9$  HV).

HEAT 2, austenitised at 1130°C, 1150°C or 1175°C, followed by oil quenching, sub-zero treatment at -196°C and tempering at 550°C, displays final hardness values between 626 HV and 644 HV. The microstructures of samples austenitised at 1130°C and 1150°C are shown in Figures 4.30 and 4.31. These samples are shown to consist of martensite, small amounts of retained austenite and carbides. Retained austenite contents of 1% (austenitised at 1130°C) and 3% (austenitised at 1150°C) were measured. A scanning electron micrograph of HEAT 2 after austenitising at 1175°C, followed by oil quenching, sub-zero treatment and tempering at 550°C, is shown in Figure 4.32. This microstructure consists of martensite, approximately 4% retained austenite and a network of reprecipitated grain boundary  $M_{23}C_6$  carbide particles. A structure with a pronounced grain boundary network of carbides does not satisfy the requirement of evenly dispersed carbides, as stated in Chapter 2.

A temper temperature of 700°C proved to be excessive for both heats, resulting in extensive softening. Tempering at this temperature after sub-zero treatment yielded hardness values well below 400 HV in both heats. A representative micrograph of HEAT 2, austenitised at 1130°C, sub-zero treated at -196°C and tempered at 700°C, is shown in Figure 4.33.



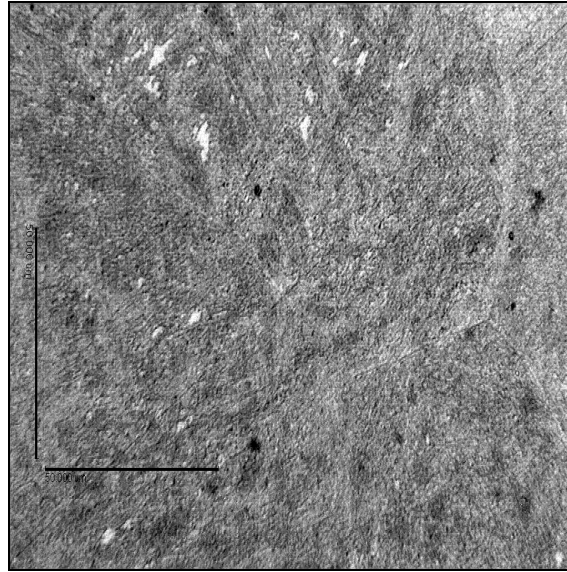
**Figure 4.30:** Optical micrograph of HEAT 2 after austenitising at 1130°C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 550°C. The microstructure consists of martensite and 1% retained austenite. (Hardness: 626±4 HV). (Magnification: 200x).



**Figure 4.31:** Scanning electron micrograph of HEAT 2 after austenitising at 1150°C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 550°C. The microstructure consists of martensite and 3% retained austenite. (Hardness: 644±9 HV).



**Figure 4.32:** Scanning electron micrograph of HEAT 2 after austenitising at 1175°C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 550°C. The microstructure consists of martensite, 4% retained austenite and networks of intergranular  $M_{23}C_6$  carbides. (Hardness: 647±12 HV).



**Figure 4.33:** Optical micrograph of HEAT 2 after austenitising at 1130°C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 700°C. The microstructure consists of martensite and retained austenite. (Hardness: 333±2 HV). (Magnification: 500x).

A summary of the effect of sub-zero treatment and tempering on the hardness of HEATS 1 and 2 is shown in Table 4.4. It is evident that austenitising at temperatures between 1130°C and 1175°C, followed by oil quenching, sub-zero treatment in liquid nitrogen and tempering at 550°C, satisfy the hardness requirements stated in Chapter 2.

**Table 4.4:** The effect of sub-zero treatment and tempering on the hardness of HEATS 1 and 2 after austenitising at 1130°C, 1150°C and 1175°C.

HEAT 1				
Austenitising temperature	As-quenched	Sub-zero treated	Tempered at 550°C	Tempered at 700°C
1130°C	474±7 HV	739±1 HV	673±6 HV	372±7 HV
1150°C	308±6 HV	700±15 HV	651±14 HV	380±4 HV
1175°C	279±4 HV	702±16 HV	649±9 HV	372±7 HV

HEAT 2				
Austenitising temperature	As-quenched	Sub-zero treated	Tempered at 550°C	Tempered at 700°C
1130°C	620±4 HV	741±18 HV	626±4 HV	333±2 HV
1150°C	609±10 HV	756±9 HV	644±9 HV	330±3 HV
1175°C	488±3 HV	751±16 HV	647±12 HV	326±6 HV

#### **4. PRELIMINARY CONCLUSIONS**

The results of this investigation therefore indicate that it is possible to obtain a martensitic structure with minimal retained austenite, evenly dispersed, spherical carbides, and hardness values between 610 HV and 740 HV in both heats of medium-carbon AISI 420 martensitic stainless steel supplied for the purpose of this investigation. The following heat treatment procedure is recommended to ensure these properties:

- Austenitise at temperatures between 1130°C and 1150°C.
- Oil quench.
- Sub-zero temper in liquid nitrogen at -196°C.
- Temper at 550°C for 1 hour.

The final structure is predominantly martensitic, with minimal retained austenite (between 3% and 5%) and evenly dispersed carbides. Austenitising at temperatures higher than 1150°C gives rise to the formation of a distinct grain boundary network of  $M_{23}C_6$  carbides during tempering, and should be avoided.

The final conclusions and recommendations are discussed in Chapter 5.

## **CHAPTER 5 – FINAL CONCLUSIONS AND RECOMMENDATIONS**

### **1. FINAL CONCLUSIONS**

Medium-carbon AISI 420 martensitic stainless steel is recommended for use in applications requiring moderate corrosion resistance, high hardness, excellent wear resistance and good edge retention in cutting surfaces. The steel is usually supplied in the fully annealed condition, with a microstructure consisting of coarse, globular carbides in a ferrite matrix. Annealing softens the steel in preparation for further cold work or machining operations. After forming, the consumer or fabricator performs the final hardening treatments to develop optimal mechanical and corrosion properties. The microstructure and properties of medium-carbon AISI 420 martensitic stainless steel depend strongly on the hardening heat treatment, and in particular the austenitising treatment, that the steel receives. The austenitising temperature controls the partitioning of alloying elements between the austenite and carbides at elevated temperature, and affects the martensite transformation range, grain size, hardness and the retained austenite content of the steel in the as-quenched condition. This project aimed at identifying the heat treatment parameters required to produce a martensitic structure with minimal retained austenite and evenly dispersed carbides, and hardness values between 610 HV and 740 HV (hardness on the Vickers scale) in two heats of AISI 420 martensitic stainless steel. The following conclusions were drawn:

- Higher austenitising temperatures lead to increased carbide dissolution. The carbide densities in both steels decrease with an increase in austenitising temperature. The higher molybdenum content of HEAT 1 retards carbide dissolution at higher austenitising temperatures due to the increased stability of the carbides (as confirmed by phase diagrams constructed using the CALPHAD model). Complete carbide dissolution takes place at temperatures higher than 1175°C in both heats.
- The gradual dissolution of carbides at austenitising temperatures up to 1175°C (where complete carbide dissolution is observed in both heats) affects the measured retained austenite content, as-quenched hardness and grain size of the steels. The measured hardness values of both heats increase slightly with

an increase in austenitising temperature up to 1075°C. This can be attributed to the partial dissolution of  $M_{23}C_6$  carbides, which raises the carbon content of the austenite phase, as a result of which a higher carbon martensite forms on quenching.

- Higher austenitising temperatures raise the amount of carbon and alloying elements in solution in the austenite, and depress the martensite transformation range to lower temperatures. At temperatures higher than approximately 1075°C, increased carbide dissolution results in higher retained austenite contents, particularly in HEAT 1, and a considerable reduction in hardness.
- The dissolution of carbides during austenitising affects the austenite grain size. The average ASTM grain size number remains stable at well above 9 for austenitising temperatures below about 1075°C. At austenitising temperatures between 1075°C and 1200°C, the grain size increases rapidly. This increase in grain size is associated with the increase in temperature (providing a higher driving force for grain growth during heat treatment), compounded by the dissolution of grain pinning carbides.
- Tempering at temperatures of 550°C, 650°C or 750°C after austenitising at 1150°C and 1175°C does not reduce the as-quenched retained austenite content or induce secondary hardening. Significant secondary hardening is observed only after tempering at 750°C in HEAT 1, but the hardness values are well below those required. Double tempering at the same temperatures is also not effective.
- Sub-zero treatment in liquid nitrogen after austenitising at 1130°C or 1150°C raises the hardness values considerably in both HEAT 1 and HEAT 2, with final hardness values above 690 HV in all the samples evaluated. This suggests that the majority of the retained austenite in the as-quenched microstructures transforms to martensite during sub-zero treatment, resulting in a significant increase in hardness. Tempering at 550°C after sub-zero treatment results in hardness values well within the range specified. The microstructure after tempering at 550°C consists of martensite with minimal retained austenite and evenly dispersed carbides. Austenitising at temperatures higher than 1150°C



gives rise to the formation of a distinct grain boundary network of  $M_{23}C_6$  carbides, and should be avoided.

## 2. RECOMMENDATIONS

The results of this investigation confirm that it is possible to obtain a martensitic structure with minimal retained austenite, evenly dispersed, spherical carbides, and hardness values between 610 HV and 740 HV in both heats of medium-carbon AISI 420 martensitic stainless steel supplied for the purpose of this investigation. The following heat treatment procedure is recommended to ensure these properties:

- Austenitise at temperatures between 1130°C and 1150°C. Short soaking times of 15 minutes are sufficient for light sections, but the austenitising time needs to be adjusted for heavier sections.
- Oil quench.
- Sub-zero temper in liquid nitrogen at -196°C.
- Temper at 550°C for 1 hour.

The final structure is predominantly martensitic, with minimal retained austenite (between 3% and 5%), and contains evenly dispersed carbides. Austenitising at temperatures higher than 1150°C gives rise to the formation of a distinct grain boundary network of  $M_{23}C_6$  carbides, and should be avoided.