

THE EFFECT OF PRIOR TEMPERING ON CRYOGENIC TREATMENT TO REDUCE RETAINED AUSTENITE¹

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Abstract

The consensus view is that a high carbon case gives gears the best overall properties provided that there is no carbide network and that the retained austenite has been reduced below 20% by cryogenic treatment. This view is effectively enshrined in the SAE AMS 2759/7 standard. The cryogenic treatment usually takes place immediately after the quench to avoid austenite stabilisation. However, for some parts with complex geometries that might crack during the treatment, a short low temperature temper is carried out first. Little is known on how this temper affects the subsequent cryogenic treatment. Three carburising steels used extensively in the aerospace industry were carburised to produce high retained austenite levels in the case using two different, but typical carburising cycles. The retained austenite was determined by XRD before and after cryogenic treatment carried out in accordance with the standard and compared with that obtained when an intermediate temper was used. This study shows that for three typical carburising steels, carburised using typical cycles, the efficacy of the cryogenic treatment is reduced only slightly after the temper, and not enough to be industrially significant.

Keywords: Cryogenic treatment; Tempering; Retained austenite.

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1 INTRODUCTION

After carburising, a component is usually quenched to around room temperature. If the carbon content of its case is higher than a certain limit – around 0.8% for mild steel and down to around 0.65% for alloy carburising steel – then some of the high temperature austenite phase will be “retained” in the otherwise martensitic case. This retained austenite will increase in proportion to the carbon content.^[1,2] It can be converted to martensite either by continuing quenching to below room temperature – cryogenic treatment - or by tempering. Components are usually tempered in any event to reduce the brittleness of the martensite, but at a temperature that is rarely high enough to remove the retained austenite. Using a high enough temperature – typically 300°C – would, however, reduce the hardness of the case considerably. Cryogenic treatment can provide a solution - some typical reductions in retained austenite achieved by cryogenic treatment are shown in Table 1.^[3]

Table 1. Retained austenite percentage for carburised 20MnCr5 at 50 µm

	0.8% carbon	1.0% carbon
After conventional treatment	9.1	31.0
After cryogenic treatment at -70°C	3.3	13.5

Although -70°C is usually deemed low enough to convert the major part of the retained austenite to martensite it has been shown that for carburised 21NiCrMo2 and 16MnCr5 the colder the treatment the more austenite is converted.^[4] However, it is not always possible to convert all the austenite. The austenite tends to stabilize with time so it is better to carry out the cryogenic treatment as soon as possible after quenching, preferably within an hour.^[2,5]

The aerospace industry uses the SAE AMS 2759/7 standard for carburising components made from a variety of steels.^[6] This standard states that for Class 1 parts the surface carbon shall be in the range 0.7% to 1.0% and retained austenite shall not exceed 10%. For Class 2 parts the surface carbon range is 0.75 to 1.10% and the retained austenite less than 20%. A carbon potential of 0.9% was therefore selected for these tests as it is close to the middle of the range for both classes.

The standard also states that both classes of parts must be sub-zero treated at -73°C or lower. A sub-zero temperature of -100°C was selected for the tests as it both meets the standard and is typical for the industry. The standard allows the heat treater to use a “snap temper” after quenching and prior to sub-zero treatment when the design of the part and thermal stresses may result in the part cracking. For the tests a snap temper was interpreted as 149°C for 30 minutes – the maximum temperature allowed. However, many industrial practitioners do not know exactly how much this snap temper may affect the cryogenic treatment that follows it.

In 1944 Berlien^[7] stated that *steels of the nickel carburising type do not require cooling immediately after the quench but can be refrigerated even after tempering providing that the tempering temperatures have been low or in other words in the range of 300 to 350 degrees Fahr (~150 to 180°C). Refrigeration, however, must be followed by a draw. Reactions of other carburising steels such as SAE 3115 or 3120, 4320, 4615, 4620 and 4815, to the refrigeration process are similar, but to a lesser degree.* This study was set up to elucidate exactly what effect the intermediate temper has.

The aerospace industry carburises large quantities of parts mainly manufactured from three steels: SAE8620, SAE4320 and SAE9310. These three steels, or where not available their closely related European equivalents, were used in the study. Typically, one of two carburising cycles is used. The parts are either carburised and direct quenched or carburised, cooled out, then reheated and quenched. Both these cycles were therefore included in the study.

2 EXPERIMENTAL

Samples of the three steels or their European near equivalents 16MnCr5 (SAE8620), 17CrNiMo6 (SAE4320) and SAE9310 were obtained and machined into blocks suitable for retained austenite determinations. Although not exact equivalents, the European steels are used for the same applications as their SAE counterparts. The composition of the three steels is shown in Table 2.

Table 2. The chemical analysis of the steels

	C	Mn	Si	P	S	Cr	Ni	Mo
DIN16MnCr5	0.17	1.25	0.21	0.010	0.045	1.06	0.19	0.05
DIN17CrNiMo6	0.17	0.45	0.28	0.025	0.021	1.52	1.46	0.25
SAE9310	0.10	0.63	0.28	0.011	0.016	1.22	3.16	0.10

Samples of each steel were subjected to one of two carburising treatments, either:

Carburise for 4 hours at 925°C at 1% carbon potential
Cool to 850°C and hold for 1 hour at 0.9% carbon potential
Quench into oil at 70°C

or:

Carburise for 4 hours at 925°C at 1% carbon potential
Cool to 850°C and hold for 1 hour at 0.9% carbon potential
Cool out in the furnace vestibule
Reheat to 850°C and hold for 30 minutes at 0.9% carbon potential
Quench into oil at 70°C

Subsequently, two samples of each steel were either immediately tempered at 149°C for 30 minutes followed by cryogenic treatment for one hour at -100°C or immediately cryogenic treated at -100°C for one hour. All these samples were then tempered for 1 hour at 170°C.

After heat treatment a sample from each treatment was cross sectioned and metallurgically examined, A second sample was sent to Swerea IVF in Sweden where the retained austenite below the internally oxidised surface layer was determined by X-ray diffraction (XRD). The retained austenite measurements were performed with Xstress3000 equipment. Prior to the measurements the equipment was calibrated with a stress free Fe-powder, which calibrates the distance between the sample and the collimator. In this case a 3 mm collimator was used together with vanadium (V) windows. Angles for detector A were 156.4° and 130.0° and for detector B 106.1° and 80° for the ferrite and austenite peaks, respectively. Normally retained austenite above 5% is easily detected. However at lower contents the

reliability decreases and even with a longer exposure time the accuracy might still be low.

During measurement four different hkl-planes were analysed; $\{211\}\alpha$, $\{200\}\alpha$, $\{220\}\gamma$ and $\{200\}\gamma$. The diffraction peaks were treated separately and the ferrite peaks were analysed with a linear background and a Pearson VII as curve fitting algorithm. The austenite peaks were analysed with a parabolic background and Gaussian as curve fitting algorithm. The diffraction parameters during measurement are shown in Table 3.

Table 3. Diffraction parameters

Parameters	
Collimator size	3 mm
Exposure time	30-50 sec
Inclination	45°
Psi-oscillation	0
Distance	10.43mm
Phi-oscillation	±85°
No. of phi. osc.	10 - 15

3 RESULTS

The retained austenite measurements for the three steels tested are shown in Figures 1 to 3. In all three steels, whether direct quenched or cooled out and reheated, the cryogenic treatment reduces the retained austenite. “Snap” tempering prior to the cryogenic treatment slightly reduces the effectiveness of the cryogenic treatment. The effect is slightly larger for the cooled-out, reheated and quenched samples than for the directly quenched samples.

As would have been expected, the higher the alloy content of the steel, the more retained austenite was formed in the direct quenched samples. In all the steels, cryogenic treatment at -100°C was able to reduce the retained austenite to 10 to 13% whether or not the sample was tempered prior to cryogenic treatment.

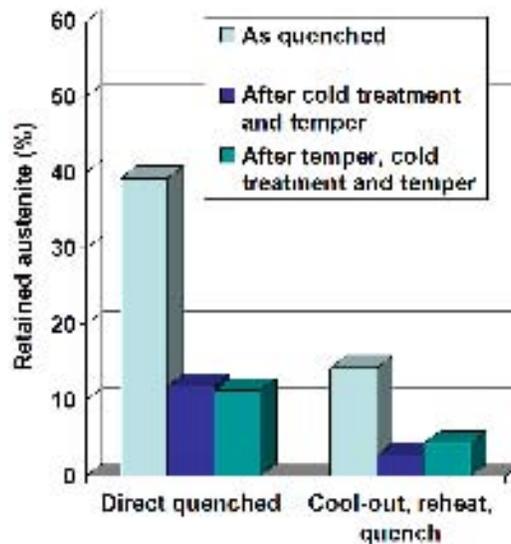


Figure 1. The retained austenite in the case of DIN16MnCr5.

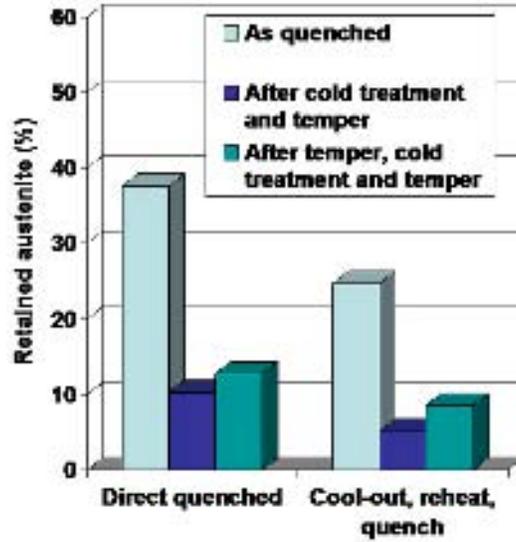


Figure 2. The retained austenite in the case of DIN17CrNiMo6.

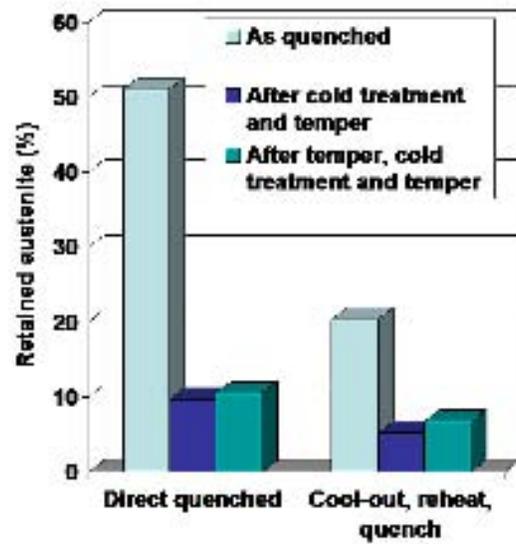


Figure 3. The retained austenite in the case of SAE9310.

Figures 4 to 6 show the hardness traverses for each of the treatments. Again as might be expected, the hardness increases after cold treatment because of the reduction in retained austenite. The samples tempered prior to cold treatment exhibit slightly lower hardnesses in line with the slightly higher retained austenite.

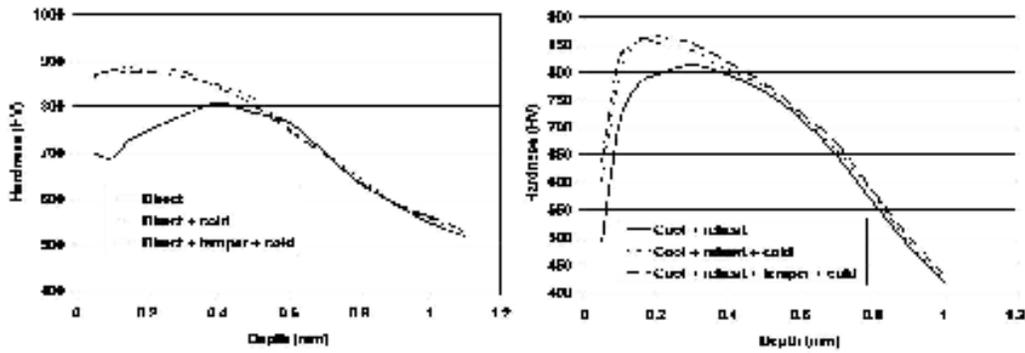


Figure 4. The hardness profiles in the case of DIN16MnCr5 before and after cold treatment.

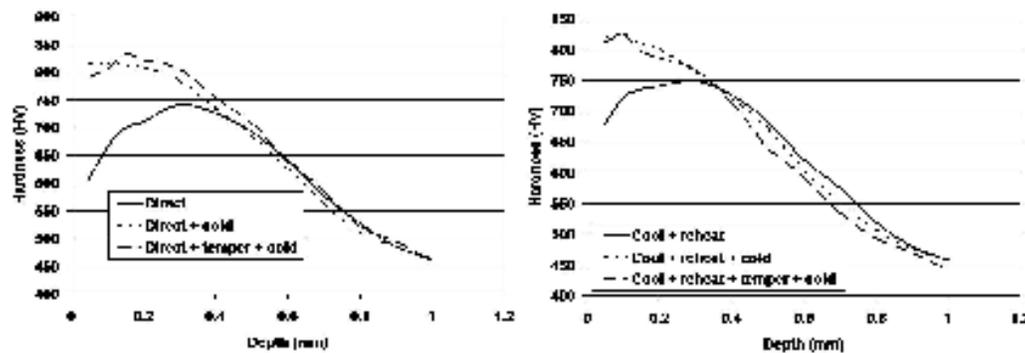


Figure 5. The hardness profiles in the case of DIN17CrNiMo6 before and after cold treatment.

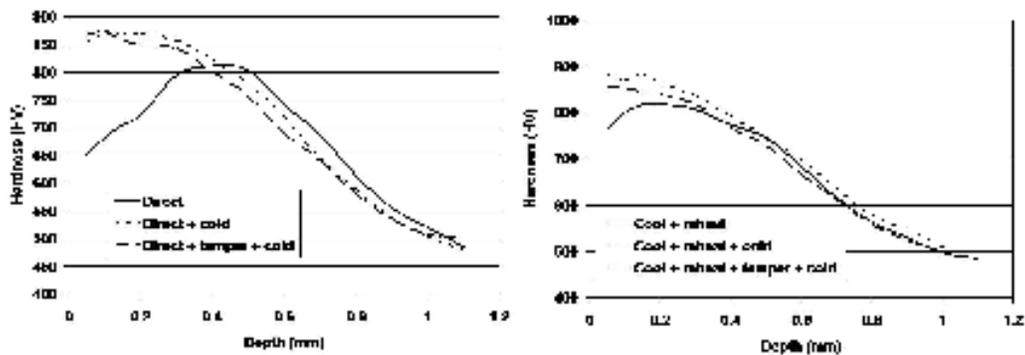


Figure 6. The hardness profiles in the case of SAE9310 before and after cold treatment.

None of the treatments gave rise to more extensive carbide networks than those allowed by SAE AMS 2759/7 Class 1 (40% of grain boundaries affected). The highest level was found in SAE9310 after cool-out, reheat and quench. The microstructure is shown in Figure 7. None of the samples showed any evidence of micro-cracking before or after cold treatment. However, given their regular shape and small size, this may have been expected.

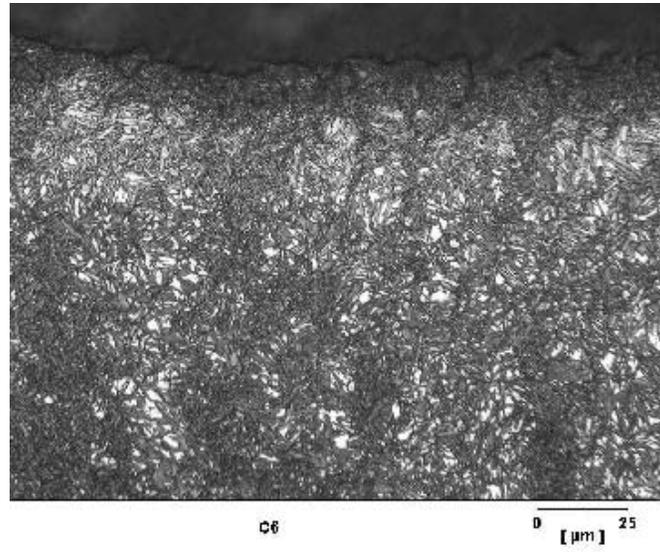


Figure 7. The microstructure of the case of SAE9310 after cool-out, reheat and quench.

Bearing in mind that the samples were treated in the same furnace load as the other steels, the very low surface hardnesses exhibited by DIN16MnCr5 after cool-out, reheat and quench (Figure 4) illustrate how susceptible this steel is to internal oxidation. DIN16MnCr5 relies almost exclusively on low levels of chromium to achieve good hardenability: once the chromium is oxidised out of solution, the hardenability falls rapidly. The resulting microstructure is shown in Figure 8.

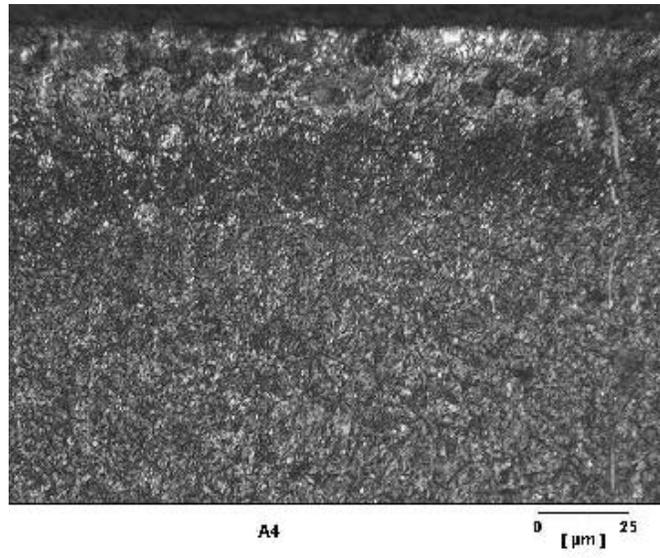


Figure 8. The microstructure of the case of DIN16MnCr5 after cool-out, reheat and quench.

The typical set of microstructures in Figure 9 show the effect of cold treatment with and without prior tempering on DIN16MnCr5. Figure 10 shows examples of the microstructure of all three steels after direct quench and cold treatment.

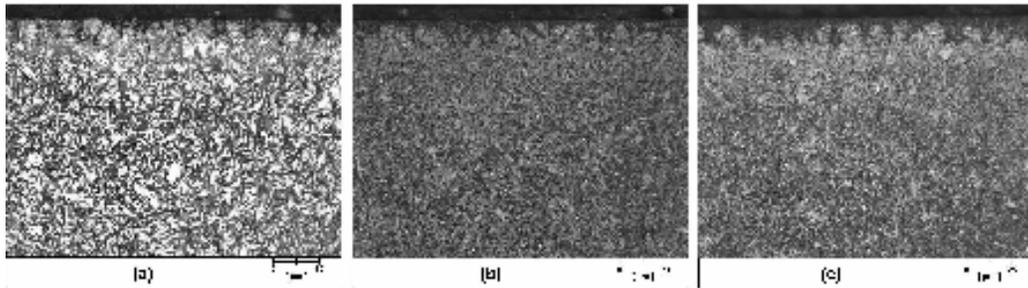


Figure 9. The microstructure of DIN16MnCr5 direct quenched after carburising: (a) as quenched, (b) quenched and cold treated and (c) quenched, tempered and cold treated.

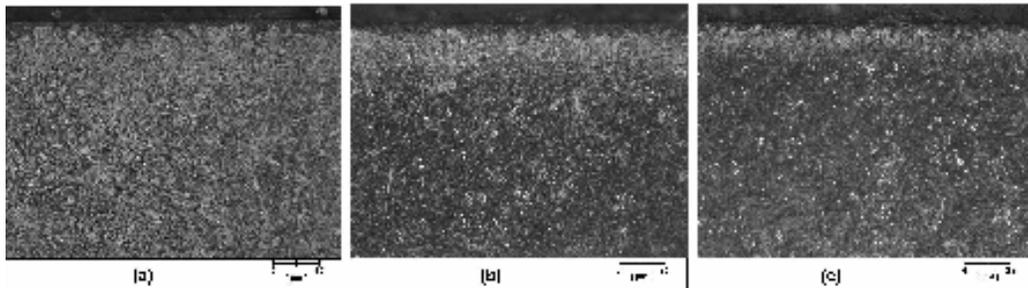


Figure 10. The microstructure of (a) DIN16MnCr5, (b) DIN17CrNiMo6 and (c) SAE9310 after direct quenching and cold treatment.

4 DISCUSSION

Even today there is no agreement in the literature with regard to tempering prior to cryogenic treatment for the removal of retained austenite. The ASM Handbook,^[8] Davis^[9] and Bowes^[10] suggest that any cryogenic treatment should be carried out prior to tempering. However, Totten et al.^[11,12] state that tempering before and after cryogenic treatment is desirable and is common practice. Aslunda et al.^[13] suggest that tempering should be eliminated entirely if maximum rolling fatigue resistance is required.

A comprehensive INFAC study using SAE9310 for the aerospace industry in 1995 reported by Cesarone in 1997^[14] did not look at the effect of tempering. It showed that, for a cycle almost identical to that used in these experiments, the retained austenite as quenched was 48.2% (by interpolation) and the retained austenite after cryogenic treatment at -78°C and tempering at 149°C was reduced to 12.9% (by interpolation). This agrees well with the data obtained in this study where 51% retained austenite was reduced to 9.7% after cryogenic treatment at -100°C and tempering at 170°C.

The author has previously shown that, for cryogenic treatment at -120°C, room temperature aging has no stabilizing effect on the retained austenite in carburised SAE8620.^[5] Imai and Izumiyama^[15] show that, for room temperature aging of a 1.12% carbon, 4.98% nickel alloy, although a lower temperature is needed to restart transformation when parts are sub-zero treated, if a sub-zero temperature of -100°C is used there is no difference in the final retained austenite whatever the length of aging. This study gives very similar results for carburised SAE9310 where the difference between samples aged for 30 minutes at 150°C and un-aged samples was within experimental error.

It may be concluded that the data obtained in this study are consistent with those in the literature, both relating to the amount of retained austenite formed during the carburising treatment and the effect of cryogenic treatment in reducing it. It may also be concluded that although the introduction of a snap temper between the quenching and the cryogenic treatment reduces makes the cryogenic treatment less effective in reducing retained austenite, the amount of the reduction is not industrially significant.

The low surface hardnesses obtained by carburising, cool-out, reheat and quench for the low alloy carburising steel DIN16MnCr5 raises some fundamental questions. The material is cheaper than its higher alloy counterparts, but the cures for its lower hardenability can be expensive. The soft layer can be removed by grinding: an expensive process and not always practical. Another option worth consideration is to use low pressure carburising where there is no internal oxidation and hence no loss of surface hardenability. However, as DIN16MnCr5 has a low general hardenability it is likely that a helium or hydrogen quench will be needed, increasing processing costs.

5 CONCLUSION

Although the introduction of a snap temper between the quenching and the cryogenic treatment reduces the effectiveness of the cryogenic treatment in reducing retained austenite, the amount of the reduction is not industrially significant.

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