HEAT TREATMENT OF CR-MO SINTERED STEELS BASED ON ASTALOY CRM

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Abstract:
For sintered steels prepared from prealloyed steel powder Astaloy CrM (Fe-3%Cr-0.5% Mo) various heat treatment techniques and their effect on properties and microstructure are described. It could be shown that steels with carbon contents between 0.2 and 0.5 mass% can be effectively hardened by oil or gas quenching, and regular martensitic microstructures are attained also after gas quenching. The quench hardness depends on carbon level, total porosity, and high temperature sintering. Tempering is recommended either in the range <200°C, for high hardness, or at 500-650°C for optimum strength / toughness. For surface hardening, low pressure carburising of sintered steels with <0.3%C, followed by gas quenching, has shown to be quite effective although carburizing temperature and carbon activity have to be carefully balanced to avoid excessive carbide formation. High sintered density, which can be obtained best by warm compaction combined with high temperature sintering, is beneficial for the formation of well defined hard surface layers with high wear and contact fatigue resistance.

1. Introduction

For low-alloy structural steels produced by ingot metallurgy, chromium, together with manganese and vanadium, is the most widely used alloy metal [1, 2]. Chromium addition results in increased hardenability and resistance to temper softening and is considerably cheaper than e.g. Ni, Mo, or Cu. In powder metallurgy, however, alloy steels mostly contain these latter elements due to their lower sensitivity to oxygen which means that these steels can be sintered in atmospheres of average quality while in the same atmosphere Cr alloying would result in intolerable oxidation. Cr prealloy steels are produced by water atomisation with subsequent carbothermic reduction in vacuum [3]; however, production volumes have been rather small. Oil atomisation [4] yielded excellent powder quality but was not maintained in industrial production. Recently, a new approach was made by Höganäs AB to introduce Cr as alloy element in PM steels [5]. The resulting new powder grade Astaloy CrM (Fe-3%Cr-0.5%Mo) is produced by water atomisation with subsequent anneal in reducing atmosphere and is available with fairly low oxygen content (typically 0.13 mass%). Compacts from this powder with about 0.2 …. 0.5% carbon have been sintered successfully in industrial furnaces if the oxygen potential in the atmosphere was sufficiently low, in which case, the dimensional and mechanical properties are very satisfactory, and esp. warm compaction combined with high temperature sintering has been outstandingly successful [6], indicated e.g. by Charpy impact values of up to >60 J. Cr alloy steels based on Astaloy CrM thus can be regarded as at least a match for the classical Ni-Cu-Mo steels.

However it must be considered that using these new Cr alloy steels as-sintered does not fully exploit their potential. As stated above, Cr results in excellent heat treatment properties (for sinter hardening shown e.g. in [7]), and it can be expected that both by quench-and-temper treatment and by carburizing, significant improvement compared to the as-sintered state should be obtainable. Within this work, the response of sintered steels based on Astaloy CrM to both through and case hardening has been studied.

2. Experimental procedures

Base steel powder Astaloy CrM was mixed with varying amounts of carbon and 0.8% EBS lubricant. The mixes were uniaxially compacted to form impact test bars 55 x 10 x 10 mm^3, the compacting pressure being commonly 700 MPa, for case hardening being varied between 200 and 800 MPa. In part specimens were also produced by warm compacting at about 130°C [8]. Sintering was done in a pushtype furnace with Mo heating elements at 1120°C and 1250°C, respectively. The atmosphere was N_2-10%H_2 of technical purity; in order to avoid oxidation/decarburization, getter boxes with getter Fe8Al-Al_2O_3 were used.

For through hardening by oil quenching, the as-sintered bars were austenitized in a pusher furnace in high purity N_2 (99.999%). Austenitizing was done for 30 min at 850 to 1000°C, followed by oil quenching. The hardness HV30 was measured in cross sections. Tempering was done in the same furnace and atmosphere between 100° and 700°C. For the gas quenching tests, a 3-zone IPSEN vacuum furnace was employed; tempering was done in a laboratory furnace in air.

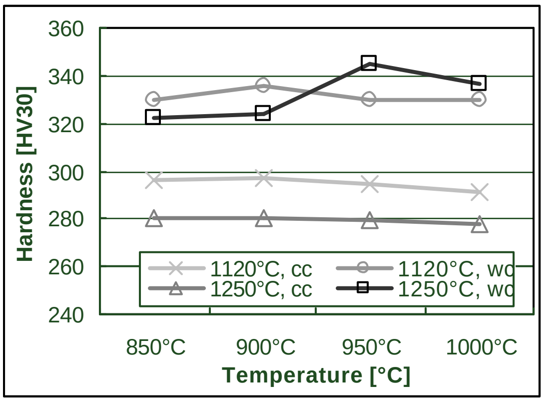
For the case hardening tests the porosity of the specimens was regarded to be of decisive importance. Therefore, impact tests bars were cold uniaxially compacted at pressures of 200, 400, 600, and 800 MPa, respectively. 0.35% and 0.5%C were admixed as graphite. As a reference, bars were also produced from Astaloy85Mo-0.3%C. The bars were sintered in flowing N_2-H_2 as described above. Low-pressure pulse carburising was done in a furnace AICHELIN at 930°C, as carburising agents both propane C_3H_8 and acetylene C_2H_2 being tested. After carburising the specimens were quenched in nitrogen. The bars were investigated metallographically, and hardness was measured at the surfaces as HV5 and in the cross section as HV0.1 profiles.

3. Experimental results

3.1. Through hardening by oil quenching

In Fig.1 the as-quenched hardness for AstaloyCrM-0.2% and -0.5%C is shown as a function of the austenitizing temperature. Evidently for both carbon levels the highest hardness is attained after quenching from 900°C, although of course the hardness differs considerably. Even at 0.2%C, however, quenching from 850°C results in reasonable hardness, indicating that the material has been fully austenitic, which is in agreement with [9]. Only in the case of warm compaction/sintering at 1250°C, austenitizing at 950°C yields slightly better hardness levels. It is also noteworthy that when sintering at 1250°C the difference in resulting hardness between cold and warm compaction is more pronounced.

Since the sintering conditions affect the carbon level and thus the maximum attainable hardness, also this latter parameter has to be considered. In Fig.2a the hardness after quenching from 900°C is plotted against the combined carbon content; here it stands out clearly that despite the lower carbon levels found after sintering at 1250°C [6, 10] quite similar hardness values are attained as after sintering at 1120°C; it also can be sent that warm compaction is more beneficial if combined with sintering at 1250°C. The rather modest influence of the austenitizing temperature compared to that of the carbon content is clearly shown in Fig.2b. It can thus be concluded that for maximum hardness high density combined with reasonably high sintering temperature is desirable (although the higher carbon loss has to be compensated for); the austenitizing temperature, on the other hand, is of secondary importance.



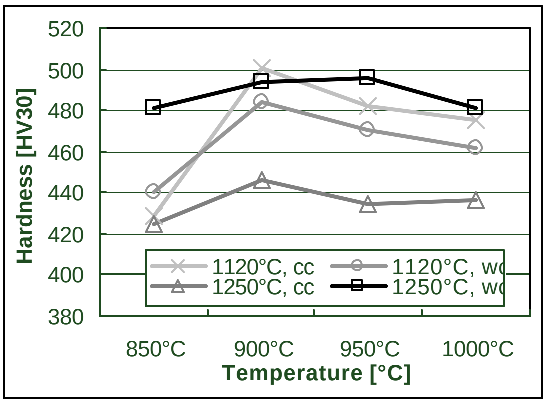
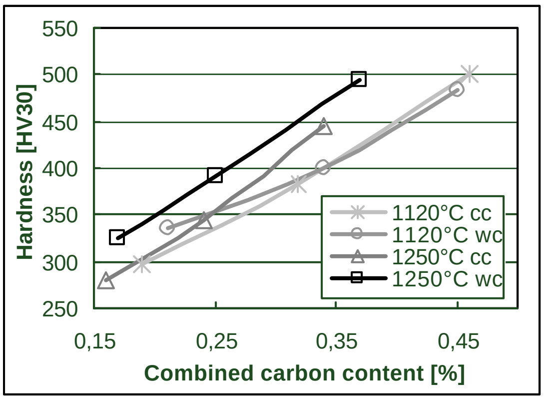


Fig.1a: AstaloyCrM-0.2%C **Fig.1b:** AstaloyCrM-0.5%C

Fig.1: Oil-quenched hardness of AstaloyCrM-x%C as a function of the austenitizing temperature. Compacted at 700 MPa, sintered 30 min 1120°C/60 min 1250°C in N_2-10%H_2.



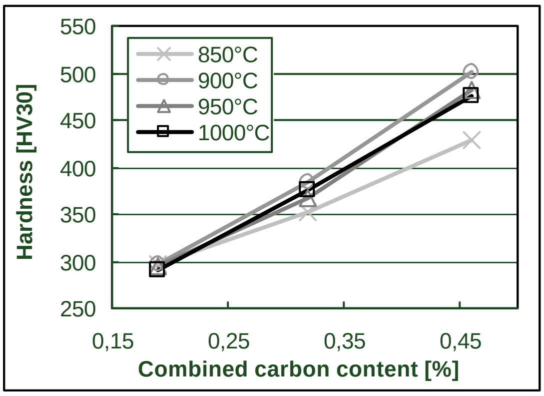


Fig.2a: Quenched from 900°C **Fig.2b:** cold compacted, sintered at 1250°C

Fig.2: Oil-quenched hardness of AstaloyCrM-x%C as a function of the combined carbon content. Compacted at 700 MPa, sintered 30 min 1120°C/60 min 1250°C in N_2-10%H_2.

3.2. Through hardening by gas quenching

For sintered components, especially for those that still contain open porosity, oil quenching is a rather messy process, gas quenching being much cleaner. The hardness values obtained by gas quenching in the IPSEN industrial vacuum furnace and by subsequent stress relieving/tempering are given in Table 1 and are compared to the hardness levels obtained by oil quenching and stress relieving/tempering. It stands out clearly that gas quenching results in almost as high hardness levels as does oil quenching, and even higher ones in the case of tempered specimens. This was also supported by the martensitic microstructure found in the gas quenched steel specimens.

Table 1

Hardness HV30 of AstaloyCrM-0.5%C admixed, differently compacted and sintered, gas quenched *(values in brackets and italics: respective hardness for oil quenching)*

State	**Cold compacted 30 min 1120°C**	**Cold compacted 60 min 1250°C**	**Warm compacted 60 min 1250°C**
As-sintered	226	248	274
Gas quenched	327 *(501)*	414 *(446)*	461 *(496)*
Q + stress relieved 150°	324 *(475)*	414 *(422)*	453 *(450)*
Q + stress relieved 200°	330 *(431)*	391 *(393)*	429 *(419)*
Q + tempered 500°	315 *(314)*	342 *(316)*	347 *(332)*
Q + tempered 580°		286	286

3.3. Case hardening

For case hardening, the carbon activity at the surface can be markedly higher than is the case in through hardening, and for steels containing strong carbide formers, carburising can result in formation of coarse alloy carbides e.g. at the grain boundaries. In order to assess the sensitivity of AstaloyCrM towards this phenomenon, the first carburising runs were performed at standard conditions, i.e. a temperature of 930°C and respective carbon activities. The hardness profiles attained for the differently compacted specimens are shown in Fig.4, Astaloy85Mo-0.3%C being also shown as a reference.

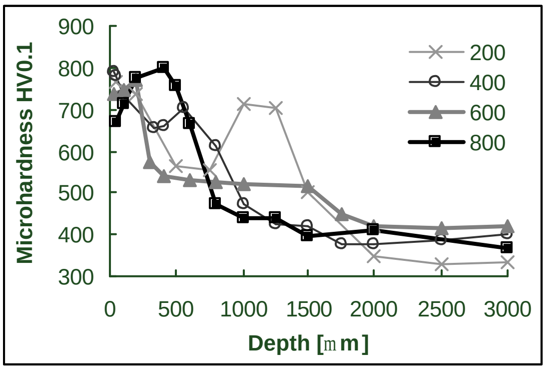


Fig.4a: AstaloyCrM-0.35%C, propane

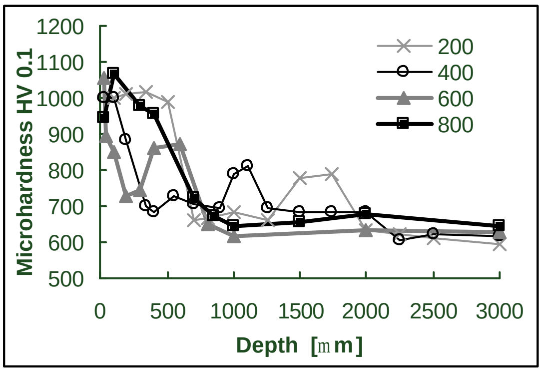


Fig.4b: AstaloyCrM-0.5%C, propane

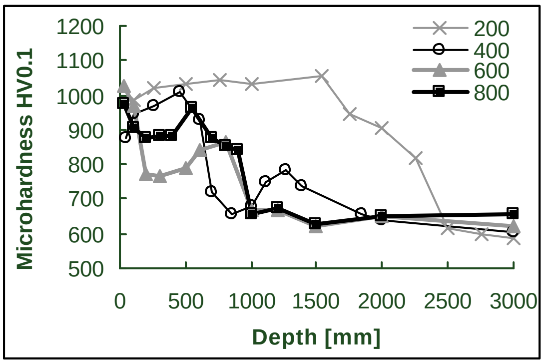


Fig.4a: AstaloyCrM-0.5%C, acetylene

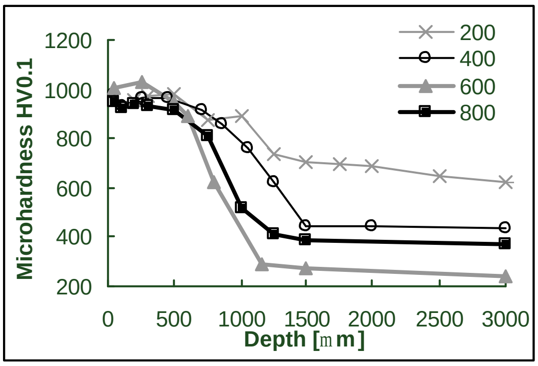


Fig.4b: Astaloy85Mo-0.3%C, propane

Fig.4: Microhardness profiles (HV0.1) of AstaloyCrM-x%C and Astaloy85Mo-0.3%C, differently compacted, low-pressure pulse carburized at 930°C and N_2 quenched

Generally it can be stated that lower density results in deeper carburisation, which phenomenon has been well known for a long time. However, even at very low density levels – at 200 MPa compacting pressure the density being in the range of 5.8..5.9 g.cm^{-3} - real through carburization – which in this case happened with standard carburizing techniques - was not observed, which indicates that low pressure carburizing is really a technique well suited for porous components.

It was also observed that in general acetylene more strongly carburizes the interior of the specimens than does propane, i.e. for formation of a defined case depth in PM components propane seems to be more suitable. This is in agreement with the findings described in [10] that acetylene is well suited for carburizing e.g. deep blind holes etc; this is exactly the type of carburizing that is not desirable with porous PM components. The difference is however most pronounced at low density; for specimens compacted at 600 or 800 MPa it is rather marginal.

If the hardness profiles in the AstaloyCrM based materials are compared to those found in Astaloy85Mo or in other sintered steels a typical irregularity in the former becomes evident: while standard sintered steels exhibit the typical profile with the hardness continuously decreasing from the surface to the core, in the AstaloyCrM based steels, at least in those with low to moderate density, there is the hardness maximum not immediately at but slightly below the surface, and a second hardness maximum is found at depths in part exceeding 1000 µm. The hardness of this second maximum in some cases is >150 HV0.1 higher than of the intermediate minimum. This phenomenon is somewhat surprising since it should be expected that the carbon activity should consistently decrease from the surface to the core, and the hardness should follow this profile. An intermediate maximum should be most improbable.

Metallographic investigations showed that under the conditions given here, apparently formation of alloy carbides has taken place in AstaloyCrM but not in Astaloy85Mo. In particular in those Cr-alloy steels with lower density, the surface contains a large proportion of carbides – cementite $(Fe,Cr)_3C$, according to [11] - , this layer being up to >500 µm thick. Below this layer, the alloy carbides have been formed mainly at the pore and grain boundaries, while the cores of the grains are comparatively soft, resulting in the marked drop in hardness observed here. Only at still larger depths the alloy carbides are no more visible, and here martensitic hardening has occurred as intended which apparently accounts for the second hardness maximum. In the high density materials the excessive carburisation at the surface did not occur; however also here alloy carbides have been formed at the grain boundaries at least near the surfaces.

In Fig.5, microstructural profiles are shown for steel specimens compacted at 400 and 800 MPa, respectively. The differences between the materials / the microstructural zones are clearly visible, and it can be seen that the microstructure in the high density materials close to the surface is similar to that in the low density ones at a depth of about 200….300 µm. the carbide-rich surface area being missing in the former material.

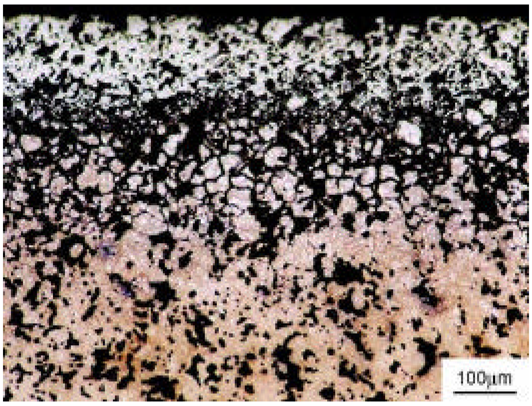


Fig.5a: Compacting pressure 400 MPa

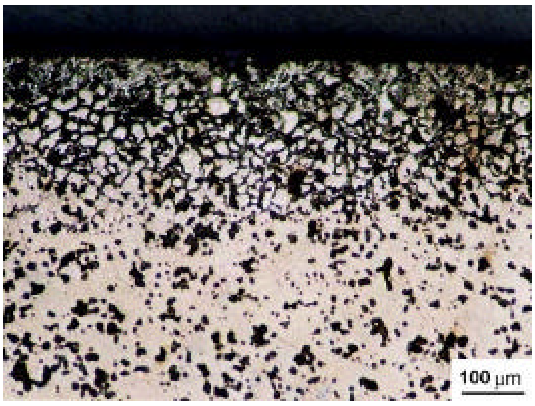


Fig.5b: Compacting pressure 800 MPa

Fig.5: Microstructure of low pressure carburized AstaloyCrM-0..%C with varying density. Sintered at 1250°C, low pressure pulse carburized at 930°C with C_3H_8, N_2 quenched

It can thus be concluded that in AstaloyCrM, formation of carbides is enhanced by the high carbon activity present during pulse carburizing and by low density, the open pores acting as transport channels for the carburizing agents. The large specific surface results in rapid overcarburizing of the surface regions which at the end of the cycle apparently consist more of carbides than of austenitic phase. At higher density levels and predominantly closed porosity this excessive overcarburizing does not occur although also here carbides are precipitated at the boundaries. According to thermodynamic calculations [8] the solubility of carbon in AstaloyCrM at 930°C is about 0.85 mass% which is in good agreement with experimental data in [11]. Apparently in highly porous components the high carbon activity during the pulsed carburizing process results in such pronounced carbide formation that the subsequent diffusion period is not sufficient to dissolve these carbides; possibly the small uncarburized volume in the test bars used here is too little of a "carbon sink" to dissolve the huge amount of carbide present in the surface zones.

4. Conclusions

- Sintered steels prepared from Cr-Mo prealloy powder AstaloyCrM and containing 0.2…0.5%C can be effectively hardened both by oil and gas quenching.
- For the oil quench hardness, the relevant parameters are the density and the combined carbon content while the austenitizing temperature is of secondary importance.
- Maximum hardness is attained at high density combined with reasonably high sintering temperature; however the higher carbon loss occurring at high temperature sintering has to be compensated for.
- After stress relieving at 150...200°C, gas quenched specimens sxhibit lower hardness levels than oil quenched ones in the case of cold pressing/sintering at 1120°C; in case of sintering at 1250°C both after cold and warm pressing, gas and oil quenched steels are well comparable. After tempering at 500°C, the gas quenched specimens are generally harder.
- Low pressure carburizing at 930°C results in pronounced carbon pickup with low density specimens and very thick case layers that consist mostly of carbides; however, real through carburizing was not observed. At higher density, the carburized layers are markedly thinner although also here grain boundary carbides can be found.
- Acetylene results in thicker case layers and more carbides being formed that does propane; the latter seems to be more recommendable for PM steels with open porosity.
- Generally, PM steels prepared from AstaloyCrM are well suited for heat treatment; for carburizing treatment, the parameters have to be carefully adjusted.

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