Heat Treatment of Distaloy AQ – Processing Aspects and Properties Achievable

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Abstract

Distaloy AQ is a lean alloyed (Fe-0.5Ni-0.5Mo) cost effective alternative to the PM steels traditionally used for heat treatment applications; these are materials that often include copper, nickel, or molybdenum in quantities of totally 2-6%. Compared to the traditional materials the low alloying content of Distaloy AQ offers relatively low hardness in sintered state, allowing for excellent as-sintered machinability. After heat treatment, however, the material performs on the same level as the more highly alloyed materials.

Performance of Distaloy AQ after different heat treatments, such as quench and tempering and case carburizing is investigated and compared to performance of materials traditionally used in heat treatments. The comparisons show on possibilities and limitations with the lean alloying route, and indicate application windows where the lean alloying route can be a competitive alternative to the traditional more costly high alloyed materials.

Keywords: Case carburizing, Quench and tempering

Introduction

Alloying elements commonly used in materials intended for heat treatment are molybdenum, nickel, and copper. The fluctuations in raw material prices for these elements have in recent years been relatively volatile, making material cost predictions difficult. Distaloy AQ is a lean alloyed material with composition is Fe-0.5Ni-0.5Mo, where the base powder is pure iron, and Ni and Mo have been diffusion bonded. The alloying content of Distaloy AQ makes it less sensitive to price fluctuation and ensures a robust product from an economical point of view.

Diffusion alloyed materials were introduced in order to provide segregation free materials with good compressibility. The alloying process consists of mixing fine elemental additions with a compressible base iron powder. The mix then undergoes a secondary thermal process in which the alloying elemental additives are bonded to the base iron by diffusion. The incomplete diffusing of the alloying elements into the iron particle is sufficient to bond the additives, but at the same time preserve the compressibility of the base iron particle. Diffusion alloying is a technology that is a patented robust process of Höganäs AB and has been consistently preferred by customers for quality.

The low alloyed route means less alloying elements for hardenability, but by using the Distaloy process, strengthening from alloying elements is concentrated to the sintering necks, where it is most needed. The result after a standard sintering process is that there will be a relatively high alloyed network in sintering necks and around original base powder particles. The interior of the largest base powder particles will be unalloyed by substitutional alloying elements but with a raised level of the interstitial element carbon due to thermodynamical coupling between C and primarily Ni. The alloying content in the network of sintering necks will at sufficient cooling rate be high enough to create a strong martensitic structure, and the net effect is higher strength compared to a pre-alloyed material with the same composition.

The alloying level of Distaloy AQ gives good machinability and sizing properties in sintered state, but still provides enough hardenability for high strength after heat treatment; a very favorable combination of properties as generally some machining is necessary in the soft state. This should be compared to traditional materials for heat treatment, with relatively high alloying content, where martensite formation already after sintering complicates sizing and machining operations.

In this study case carburizing of Distaloy AQ was performed. Case carburizing of porous specimens can be difficult; open porosity leads to penetration of the carburizing atmosphere, and control of the case depth becomes problematic. Focus of the study was on the surface hardness profile formation and the effect on the profile when heat treatment parameters were changed. Results are presented for one-dimensional diffusion in massive specimens and compared with results for tensile test bars to visualize the mass effect. As will be shown, the low alloying content of Distaloy AQ provides an extra case effect, and might therefore be a suitable material choice for case carburizing.

Experimental

The base powder used in this investigation was Distaloy AQ. It is a water atomized iron powder, with 0.5% Mo and 0.5% Ni diffusion alloyed.

Powder mixes to investigate performance after case carburizing were made with different amount of synthetic graphite (F10), and 0.6% lubricant (Table 1).

Table 1. Powder mixes used in the investigation.

Base powder	Graphite, F10 (%)	Lubricant (%)
Distaloy AQ	0.17	0.6
Distaloy AQ	0.32	0.6
Distaloy AQ	0.24	0.6

Compaction was performed to achieve green densities of 7.1 and 7.3 g/cm^3. Specimens used in the investigation were tensile test bars (ISO 2740), impact energy test bars (ISO 5754), and cylindrical specimens with a diameter of 30 mm, and a height of 30 mm. The specimens were sintered in a mesh belt furnace at 1120°C for 30 min in an atmosphere of 95/5 N_2/H_2.

The specimens were then heat treated at a subcontractor. Different carburizing times (20-60 min), carburizing temperatures (880-960°C), and carbon potentials (0.8-1.2%) were applied. To achieve desired carbon potential, addition of methanol was used. Quenching was performed in oil at a temperature of 60°C. The specimens were subsequently tempered at 200°C for 1 h in air.

A problem when heat treating sintered porous specimens is that common practice is to introduce carburizing gas already during heating. For non-porous specimens this has a negligible effect, since carburization time usually is much longer, but for sintered porous specimen this might significantly increase the carburization. In this study, carburizing was performed only at carburizing temperature, i.e. no carburizing gas was introduced during heating.

Design of experiment (DOE) methodology was used to plan and evaluate the experiments. Modeling of the data was performed with the computer software Modde [1]. From the model, each material property is described by a polynomial based on the independent variables in the investigation (density, carburizing time and temperature etc.). Properties are then predicted from this model. Predicted data might not exactly match the measured values, but with a good model relevant estimation is obtained. An advantage with this method is that a reduced trial matrix can be used and yet form a model with high accuracy. In Table 2 the reduced matrix for the case carburizing trials can be found.

Table 2. Trial matrix, mixes and heat treatments.

Experiment	**Sintered C (%)**	**Green density (g/cm^3)**	**Carburizing time (min)**	**Carburizing temp. (°C)**	**C-potential (%)**
1	0.15	7.1	20	880	1.2
2	0.3	7.1	20	880	0.8
3	0.15	7.3	20	880	0.8
4	0.3	7.3	20	880	1.2
5	0.15	7.1	60	880	0.8
6	0.3	7.1	60	880	1.2
7	0.15	7.3	60	880	1.2
8	0.3	7.3	60	880	0.8
9	0.15	7.1	20	960	0.8
10	0.3	7.1	20	960	1.2
11	0.15	7.3	20	960	1.2
12	0.3	7.3	20	960	0.8
13	0.15	7.1	60	960	1.2
14	0.3	7.1	60	960	0.8
15	0.15	7.3	60	960	0.8
16	0.3	7.3	60	960	1.2
17	0.22	7.2	40	920	1
18	0.22	7.2	40	920	1
19	0.22	7.2	40	920	1

Results and discussion

As-sintered properties

As-sintered performance has been reported earlier [2]. Performance is illustrated in Fig. 1 for two compaction levels. Corresponding densities are approximately 7.1 and 7.3 g/cm^3 respectively. The material performs at about the same level as Fe-2Cu-C. The relatively low as-sintered hardness and strength makes the material an excellent choice when sizing and/or machining prior to heat treatment is necessary. Materials conventionally used in heat treatment processes have higher alloying content, which will induce martensite formation already after sintering, which in turn makes them difficult to machine.

Through hardened properties

Through hardening will increase strength and hardness significantly. Fig. 2 shows performance after through hardening. As has been shown earlier [2,3], through hardened performance is on the same level as for more highly alloyed materials conventionally used for heat treated applications. The figure clearly illustrates the positive effect of high density for all properties presented. Both density and C increase hardness, and for high C-levels apparent hardness above 45 HRC has been reached. The figure also shows that for high carbon content, the strength will drop off. This is due to increased brittleness at high carbon level.

Case hardened properties

In Fig. 3 sample hardness profiles are presented. The curves are chosen as examples to illustrate the effect of heat treatment parameters and sample density and geometry on the hardness profile. The left image shows profiles for material with a density of 7.3 g/cm^3 after 20 min of carburizing at 880°C and a C-potential of 0.8, and the right image shows profiles for material with a density of 7.1 g/cm^3 after 60 min of carburizing at 880°C and a C-potential of 0.8. Both materials had a C-content of 0.15% prior to heat treatment. The image illustrates how long time and low density will allow deep penetration of C resulting in a more flat hardness profile. Typical value for a successful case hardening process is a surface hardness >700 HV0.01. This surface hardness has not been reached in the left figure, where the high density and short time has led to insufficient carburization.

The figure also illustrates a clear mass effect; the tensile strength bars are more effectively quenched than the massive Ø30x30 mm specimens.

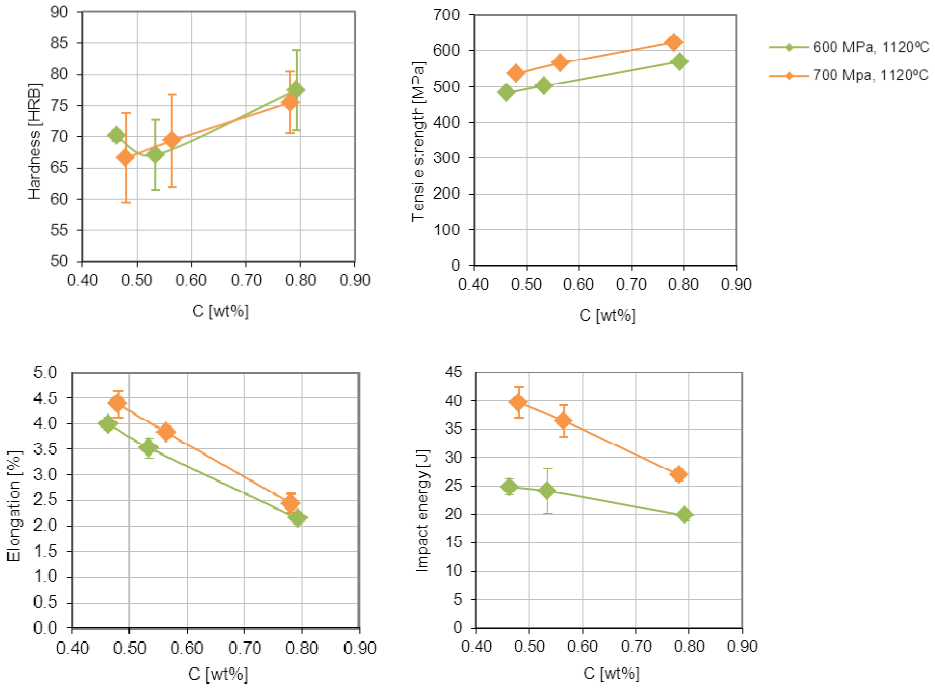


Fig. 1. Mechanical performance of Distaloy AQ after sintering at 1120°C. After [2].

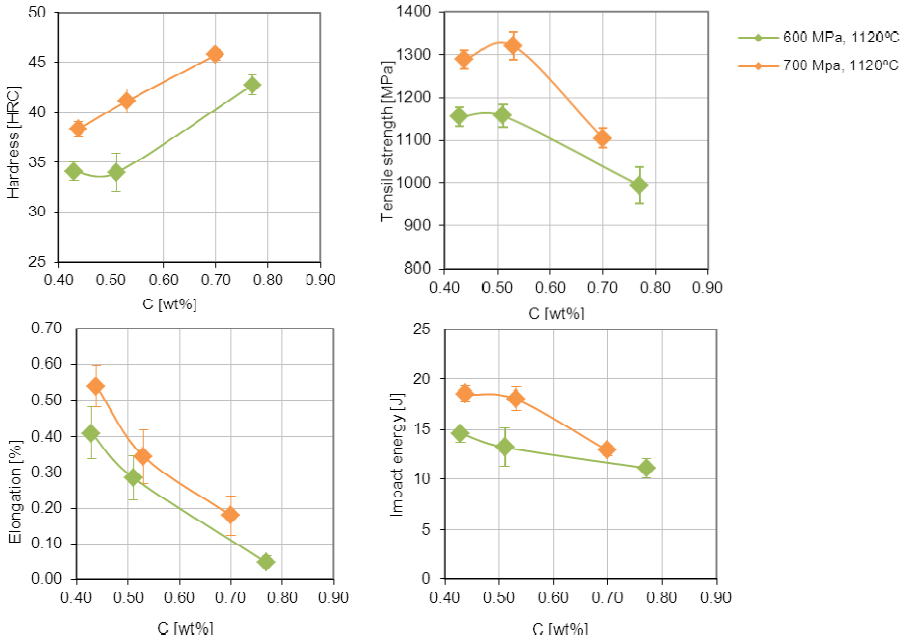


Fig. 2. Mechanical performance of Distaloy AQ after sintering at 1120°C and C-neutral quench and tempering. After [2].

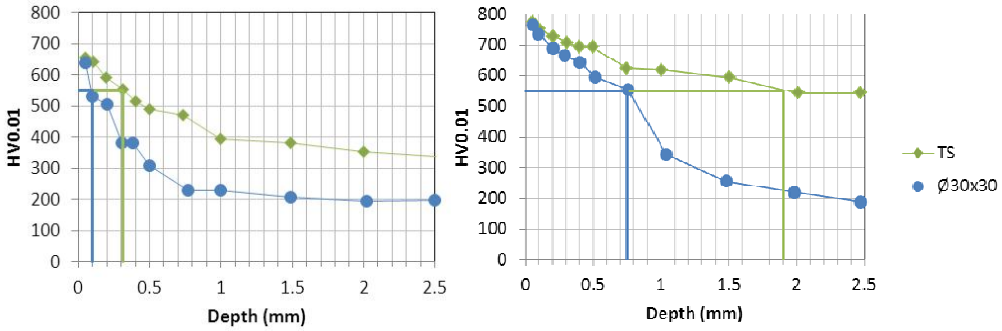


Fig. 3. Hardness profiles after case carburizing; 20 min, 0.8 C-potential, 880°C (left), 60 min, 0.8 C-potential, 880°C (right).

In the figure below (Fig. 4) typical microstructure after case carburizing are shown. The structures were obtained from a Ø30x30 mm specimens after 20 min with 0.8 C-potential at 880°C (profile presented in Fig. 3). The surface is martensitic, and the amount of fine bainite increases with depth. The bright areas are Ni-rich regions. In the core the structure is a coarse bainite with some martensite.

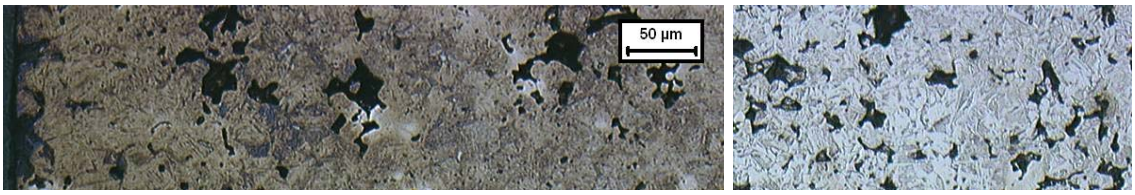


Fig. 4. Light optical micrographs of etched microstructure from surface (left), and core (right) of case carburized Distaloy AQ. Both micrographs have the same magnification.

Based on the results from the hardness profiles, modeling of the surface hardness and case depth was performed. Fig. 5 shows the result for surface hardness of Ø30x30 mm specimens with 0.15% sintered C (prior to heat treatment). Fig. 6 shows the case depth for the same specimen.

Surface hardness values are obtained in the range 600-800 HV0.01 (Fig. 5). High C-potential and long carburizing times increase the surface hardness. Longer time and/or C-potential are necessary to reach high hardness with high density specimens. A surprising result is that increased temperature and maintained C-potential results in reduced hardness. It is reasonable to assume that the higher diffusion rate and C-solubility at high temperature should instead produce a deeper profile. The result might be an effect of variations in C-potential, where the actual C-potential at high temperature has been reduced compared to the C-potential at low temperature.

Case depth for Ø30x30 mm specimens varies between 0.1 and 0.7 mm (Fig. 6). As in the case of surface hardness, time has a large effect on the depth, but no significant variation with C-potential is observed. For tensile strength test bars, both absolute values and variations in case depth are larger. Due to the relatively flat appearance of the hardness profile in many of the TS specimens, there will be a large error when determining the case depth, which in turn will lead to a weak model. Therefore only case depth results for the Ø30x30 mm specimens were successfully modeled and presented here.

Due to the uncertainties in C-potential with varied carburizing temperature, mechanical performance levels are presented for 880°C carburizing in this study. The temperature is chosen based on the high surface hardness values obtained, and the relatively wide range for case depth obtained. 880°C is a relatively low temperature for case carburizing, especially when heat treating Mo-containing materials. Mo is a ferrite stabilizing element, and with high Mo content and low C-content, there is a risk for incomplete transformation to austenite during soaking, which will result in ferrite in the quenched structure. This is of course detrimental to the performance. Mo is also a carbide forming elements, and at low heat treating temperature there might be risk for carbide precipitation. In this study neither carbides nor ferrite regions were observed, and the 880°C heat treatment was therefore considered suitable to use for evaluation of mechanical performance.

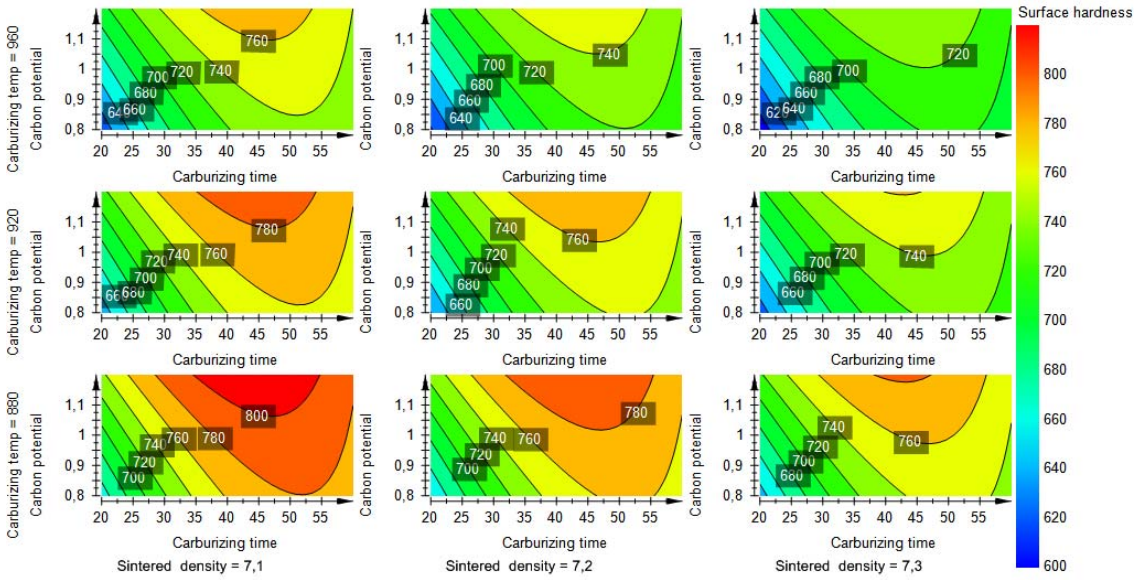


Fig. 5. Surface hardness (HV0.01) modeled for Ø30x30 mm specimens. Carbon content prior to heat treatment was 0.15%.

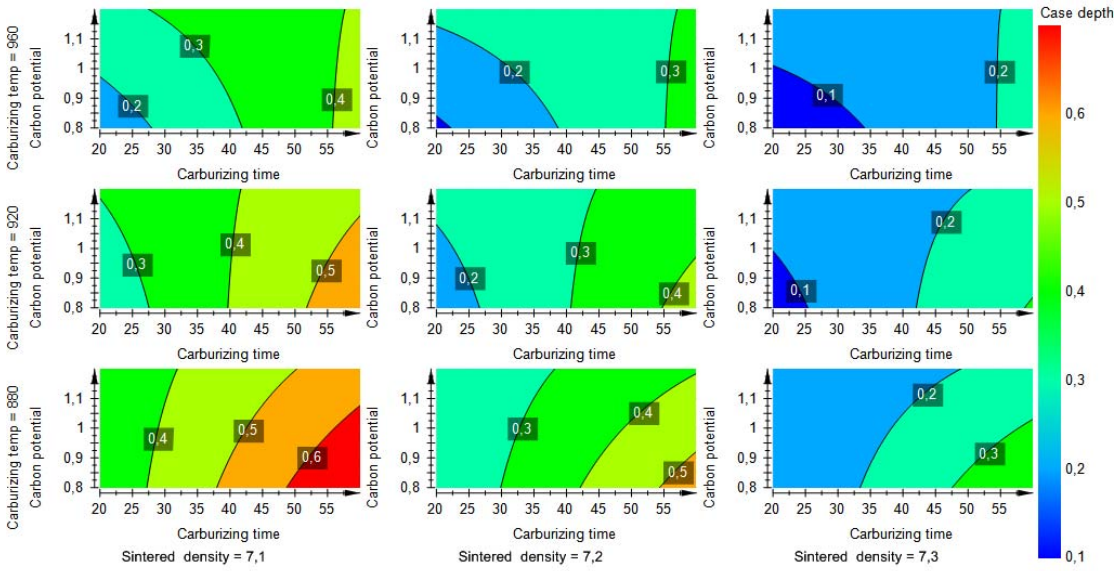


Fig. 6. Case depth (mm) modeled for Ø30x30 mm specimens. Carbon content prior to heat treatment was 0.15%.

Mechanical performance is reported in Fig. 7-Fig. 10. Carburizing was performed at 880°C, and C-content prior to heat treatment was 0.15%. In the first figure, macro hardness is presented. Values between 20-38 HRC are obtained. Hardness benefits here from a low density, since more C-diffusion is allowed, and a wider martensitic surface zone will be formed.

Fig. 8 shows tensile strength. Here bulk performance becomes more important, and a high density will therefore give a high strength. Values between 1000-1100 MPa are obtained, and the highest values are, expectedly, found for high density and high C-content.

Elongation is presented in Fig. 9, with values between 0.2-0.7%. The values are relatively low; a consequence of the large case depths. High density and low C improves elongation. It should be noted if comparison is made with as-sintered material, that the bulk microstructure is not the same. As sintered Distaloy AQ is mainly ferritic/pearlitic, while the structure in a quenched low-C material is a coarse bainite with some martensite.

Impact energy seems to have low sensitivity to heat treatment parameters (Fig. 10). Values between 10-13 J are found, and the variation for each density is of magnitude 1.5J, which is of the same order as the size of the error from analysis. The figure therefore only roughly shows what impact energy level that can be expected.

The strength and hardness obtained is lower than what is obtained for through hardened specimens, but a main reason to perform case carburizing is to increase fatigue performance. Fatigue properties have not been evaluated for the case carburized specimens in this trial, but in earlier publications competitive results for both plane bending fatigue [2,4] and gear tooth root bending [4,5] have been reported.

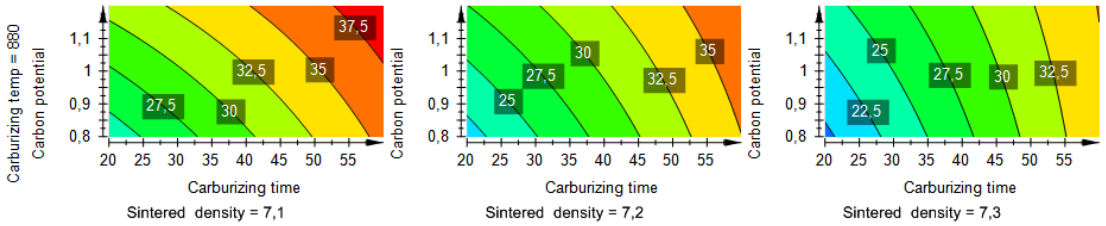


Fig. 7. Macro hardness (HRC) of tensile strength specimens after case carburizing at 880°C.

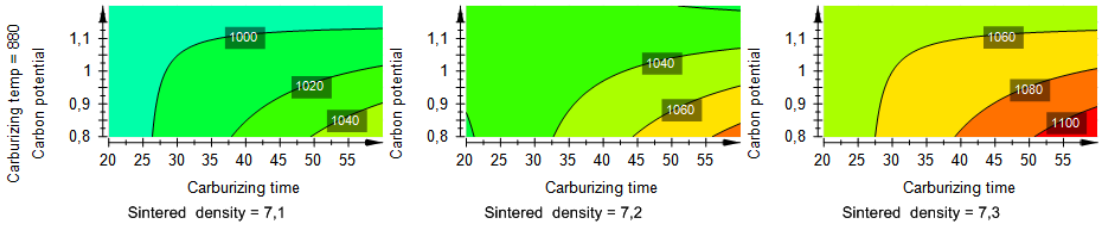


Fig. 8. Tensile strength (MPa) after case carburizing at 880°C.

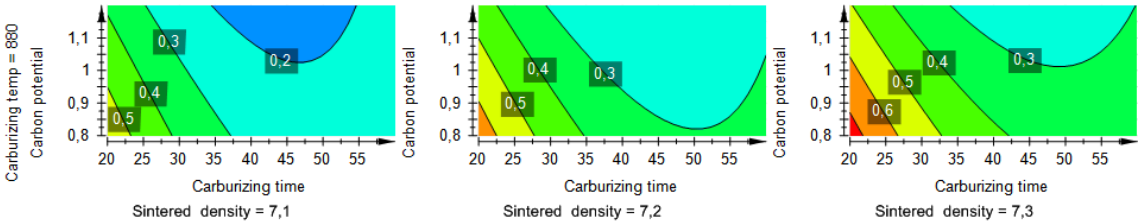


Fig. 9. Elongation (%) after case carburizing at 880°C.

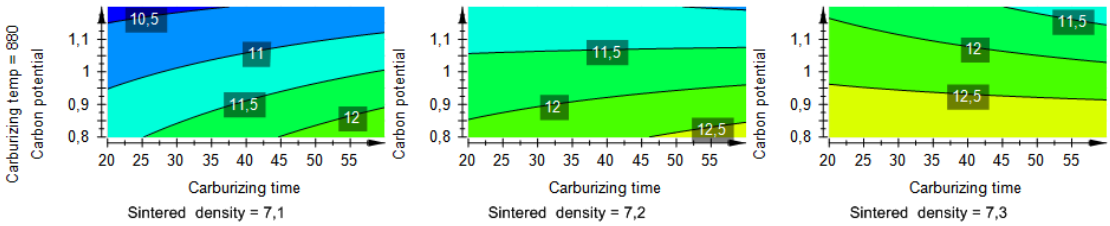


Fig. 10. Impact energy (J) after case carburizing at 880°C.

Effect of alloying system

Even though cooling rates are very high in a quenching operation, the hardenability of the material will affect the appearance of the hardness profile. Fig. 11 shows hardness profiles for four different materials and two case carburizing heat treatment processes. All materials had the same as-sintered C-content, 0.15%, and the four different materials were heat treated together, in the same batch. The materials are Astaloy CrA (Fe-1.8Cr), Astaloy Mo (Fe-1.5Mo), Astaloy 85Mo (Fe-0.85Mo), and Distaloy AQ (0.5Ni-0.5Mo). The figure shows increase both in case depth and in bulk hardness with increased alloying content and accompanying increased hardenability. Especially after

Also Astaloy 85Mo behaves as a higher alloyed material compared to Distaloy AQ, even though the total alloying content is lower. This is due to the higher content of Mo, but also due to the method of alloying. Since Astaloy 85Mo is pre-alloyed, alloying elements are evenly distributed throughout the structure, and the whole volume of the specimen experiences an increased hardenability. In the diffusion alloyed Distaloy AQ the alloying elements are concentrated to sintering necks and original particle surfaces, resulting in a lower total volume with increased hardenability due to alloying.

The relatively low alloying content of Distaloy AQ provides an extra case effect, and actually facilitates to obtain a good hardness profile. Materials with higher hardenability will get a larger case depth, especially when higher amount of carbon are allowed to diffuse into the material.

One further advantage with the low alloying content and the diffusion alloying process is the high compressibility of Distaloy AQ, which makes high densities easier to reach. As has been shown in this study, density is an important tool for good control of the hardness profile.

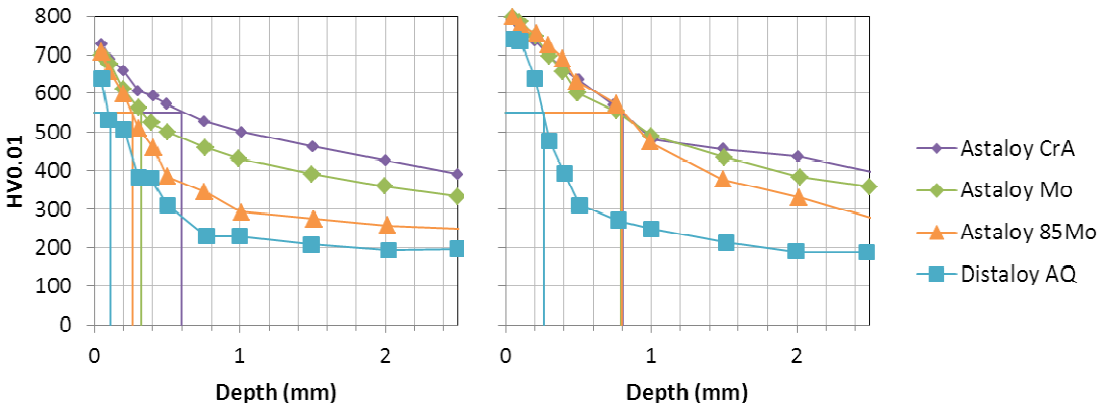


Fig. 11. Hardness profiles of Ø30x30 mm specimens with a density of 7.3 g/cm^3 after different heat treatments; 20 min at 880°C and a C-potential of 0.8 (left) and 60 min at 880°C and a C-potential of 1.2 (right). Carbon content prior to heat treatment was 0.15%.

Conclusions

- Design of experiment (DOE) methodology was used to plan and evaluate case carburizing of Distaloy AQ.
- A reduced trial matrix was utilized, and material properties could successfully be predicted by the model used.
- High sintered density significantly increases the possibility to achieve good results when case carburizing is applied.
- Surface hardness shows a high dependence on both C-potential and carburizing time.
- Case depth is mainly sensitive to carburizing time.
- Due to the low alloying content of Distaloy AQ, there is a clear mass effect when quench & tempering is performed, and case depth can be significantly larger for less massive specimens
- The low alloying content of Distaloy AQ provides an extra case effect compared to more highly alloyed materials, and makes Distaloy AQ a suitable choice for case hardening operations when not to large case depths are desired.

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