PROCESS ROUTE INFLUENCE ON PERFORMANCE OF PM STEELS PRE-ALLOYED WITH CHROMIUM

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ABSTRACT

Increased demand on high strength PM applications and cost reduction from the automotive industry has lead to introduction of chromium as an alloying element mainly due to its low cost and high hardenability. A common process for pre-alloyed material with 3% chromium has been sinter-hardening using normal sintering temperatures. Further increase in mechanical properties is achieved utilizing high sintering temperature or heat treatment operations like vacuum case hardening.

In this paper the influence of process conditions on the static and dynamic properties of materials pre-alloyed with chromium are presented and explained through their microstructure. After high temperature sintering with a carbon content of 0.7% a fatigue strength of 445 MPa (65 ksi) is obtained. Combining high temperature sintering with rapid cooling yield strength up to 1250 MPa (181 ksi) is obtained at 0.45% C. If high temperature sintering is followed by vacuum carburizing fatigue strength of 527 MPa (76 ksi) is achieved.

INTRODUCTION

A water atomized steel powder, Astaloy CrM, pre-alloyed with 3% chromium and 0.5% molybdenum was introduced some years ago [1]. Chromium as alloying elements is of interest due to its low cost, high hardenability and greater possibility to recycle, if compared to copper containing materials. Development work of this material as well as manufacturing processes have given alternatives for cost competitive production of PM parts. Demand in the PM industry for tighter tolerances makes the pre-alloyed route attractive for production of high performance parts.

In this paper the properties of high temperature sintered, Astaloy CrM based materials, are presented. The influence of carbon content and cooling rate on the static and dynamic properties of Astaloy CrM before and after a shot peening operation were investigated. Influence of high temperature sintering combined with vacuum carburising on the mechanical properties of Astaloy CrM was also studied.

EXPERIMENTAL PROCEDURE

Material

Chemical composition of the base powder used, Astaloy CrM, is shown in table I.

Table I. Composition of the base powder

	Chromium (%)	**Molybdenum (%)**	**Fe**
Astaloy CrM	3*	0.5*	Balance

* Pre-alloyed elements.

For evaluation of static and dynamic properties graphite and lubricant were admixed to the base powder. The nominal composition of the investigated mixes is reported in table II.

Table II. Admixed graphite and lubricant.

Material	**Base powder**	**Graphite (%)**	**A-wax (%)**	**Lubr. (%)**
A	Astaloy CrM	0.55	0.8	
B	Astaloy CrM	0.65	0.8	
C	Astaloy CrM	0.75	0.8	
D	Astaloy CrM	0.85	0.8	
E	Astaloy CrM	0.45		0.6%

Natural graphite (CUF-4, Kropfműl) was used. Amide wax was used as a lubricant in mixes A, B, C, D employed in the preparation of cold compacted specimen. For mix E warm compaction lubricant was used.

As-sintered properties, processing conditions

Mixes A, B, C, D were cold compacted to a density of 7.0 g/cm^3. Tensile testing bars (ISO 2740) and modified ISO 3928 fatigue bars with chamfered edges [2] were pressed. Pressing was carried out in a semiautomatic laboratory press. Sintering was carried out at 1250°C (2282°F) for 60 minutes in 90/10 N_2/H_2 atmosphere in a lifting earth furnace with no methane addition. Two cooling rates were investigated 0.8°C/s (1.4 °F/s) and 2.5°C/s (4.5 °F/s). The cooling rate refers to the temperature range 800-300°C (1672-932°F). All materials were tempered at 200°C (390°F) for 60 minutes in air except for material A cooled at 0.8 °C/s (1.4 °F/s). The materials cooled at 0.8°C/s (1.4°F/s) were shot peened with steel sand for 5 minutes.

Hardness HRC was measured on the as-sintered samples. Vickers hardness, HV10, was measured on the shot peened samples since HRC (150 kg load) would have covered a too deep area. Static properties were investigated both for the materials cooled at 0.8°C/s (1.4 °F/s) and 2.5°C/s (4.5 °F/s). Dynamic properties were investigated for the materials cooled at 0.8°C/s (1.4 °F/s).

As-heat treated properties, processing conditions

Mix E was used for investigating the mechanical properties that can be achieved with Astaloy CrM after high temperature sintering followed by carburising in vacuum. Mix E was warm compacted to a green density of 7.15 g/cm^3. The specimens were sintered at 1300 °C (2372 °F) in a laboratory vacuum furnace for 30 min. Subsequently they were cooled at 1050 °C (1922 °F). At this temperature the samples were carburised with acetylene gas for a total of 8 minutes. Thereafter, cooling was performed in a N_2 atmosphere at 2 bar pressure. In the next step, the samples were moved to another vacuum furnace, where they were heated at 950 °C for 15 min and then cooled with N_2 at 7 bar pressure. The samples were tempered at 200 °C (390 °F) for 60 min in air.

RESULT

Physical properties

Green-, sintered density, as-sintered carbon and oxygen content, dimensional change from green to as sintered were measured. The results are summarised in table III.

Table III. As sintered properties after cooling at 0.8°C/s (1.4 °F/s) and 2.5°C/s (4.5 °F/s)

Material	**Green Density (g/cm^3) (0.8/2.5°C/s)**	**Sintered Density (g/cm^3) (0.8/2.5°C/s)**	**Carbon (%) (0.8/2.5°C/s)**	**Dimensional Change (%) (0.8/2.5°C/s)**
A	7.0	7.17 / 7.18	0.40 / 0.37	-0.86/-0.92
B	7.0	7.15 / 7.16	0.50 / 0.45	-0.85/-0.93
C	7.0	7.15 / 7.14	0.58 / 0.55	-0.90/-0.89
D	7.0	7.16 / 7.15	0.69 / 0.65	-0.97/-0.95

High temperature sintering increases the density from 7.0 g/cm^3 to ~7.16 g/cm^3 due to a shrinkage of 0.86% to 0.97%. Sintered carbon is ~0.15% lower than the admixed graphite content. Oxygen content after sintering is <0.01% for all investigated materials.

Static properties, as-sintered

At a cooling rate of 0.8°C/s (1.4 °F/s) the yield strength increases when raising the carbon content from 0.4 to 0.6%, figure 1a. The largest increase in yield strength is obtained when increasing the carbon content from 0.4 to 0.5%. This is related to an increase in the martensite content, as seen in table IV. At 0.4% C the microstructure consists of 85% upper bainite and 15% martensite while at 0.5% C the microstructure consists of marteniste with 5-15% lower bainite. The hardness value at 0.4% C is below 30 HRC, figure 1b. This indicates that the material under these conditions has not taken hardening according to the definition that a material is hardened if it consists of more than 50% martensite or has a hardness value greater than 30 HRC. By raising the carbon content to ~0.5% an optimum in yield strength is achieved while the hardness (HRC) is raised from 25 to 45 units.

When raising the cooling rate from 0.8 °C/s (1.44 °F/s) to 2.5°C/s (4.5 °F/s) the the yield strength is increased. At 0.4%C the yield strength is raised from 770 MPa (112 ksi) to 1157 MPa (168 ksi). This is due to a transformation in the microstructure from mainly upper bainite (85%) to an enterely martensitic structure. Maximum yield strength is found at ~0.45%C. Increased cooling rate moves this optimum to lower carbon content [3]. Further increase in sintered carbon decreases the yield strength. At carbon content above 0.5% same hardness level (45 HRC) is achieved at both cooling rates tested.

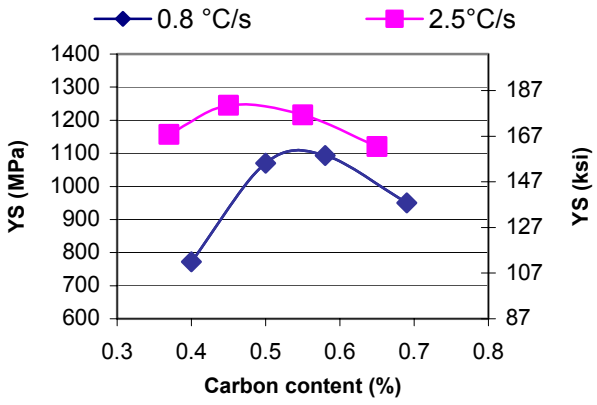


Figure 1a. Yield strength vs. carb content, at cooling rate of 0.8 °C/s (1.4 °F/s) and 2.5 °C/s (4.5 °F/s)

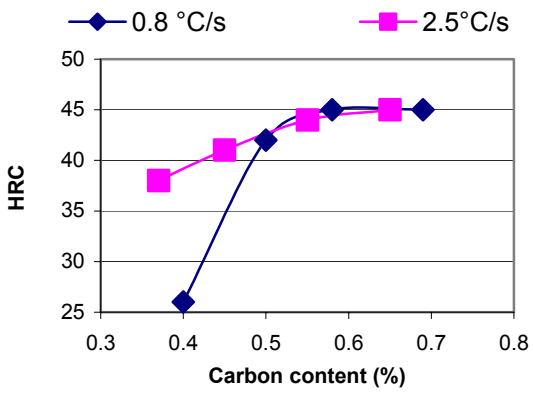


Figure 2a. HRC vs. carb content, at cooling rate of 0.8 °C/s (1.4 °F/s) and 2.5 °C/s (4.5 °F/s)

Table IV. Phase amounts (investigated on TS samples)

	0.8°C/s					2.5°C/s		
	C%	**Upper Bainite %**	**Lower Bainite %**	**Martensite %**	**Retained Austenite %**	**C%**	**Martensite %**	**Retained austenite %**
A	0.40	Bal.		15-20		0.37	100	
B	0.50		5-15	Bal.		0.45	100	
C	0.58		1-2	Bal.		0.55	100	
D	0.69		<0.5	100	Small amounts	0.65	100	Small amounts

In figure 2a CCT diagram for Astaloy CrM with 0.4%C and 0.5%C and density 7.0 g/cm^3 is shown. Corresponding phase amount measurements are shown in figure 2b. CCT diagram for 0.4% C indicates that a cooling rate > 3 °C/s (5.4 °F/s) is needed to obtain a fully martensitic structure. If the phase amounts are plotted against cooling rate the microstructure contains more than 80% martensite above 2 °C/s (3.6 °F/s). Increased carbon to 0.5% C results in a fully martesitic structure when cooling at 1°C/s (1.8 °F/s). The microstructure obtained in this investigation when cooling at 0.8 °C/s (1.8 F/s) and 2.5 °C/s (4.5 °F/s) are in good relation with what found from the dilatometer run.

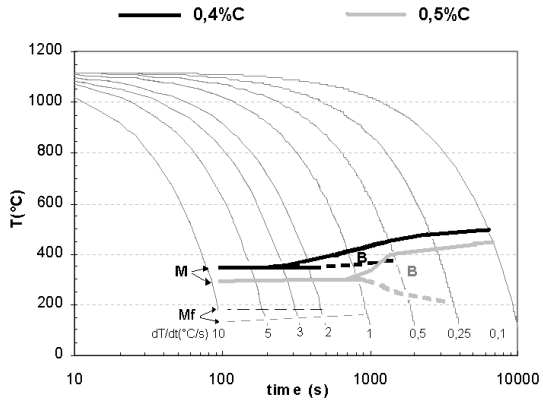


Figure 2a. CCT diagram for Astaloy CrM with 0.4 and 0.5% C.

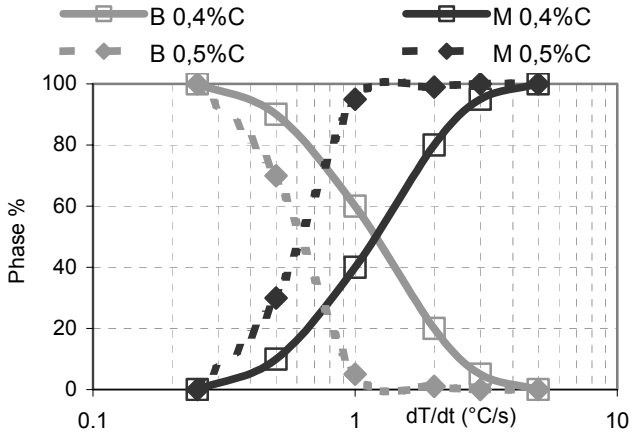


Figure 2b. Phase amount vs. cooling rate for Astaloy CrM with 0.4 and 0.5% C.

As the carbon content and cooling rate increases the elongation decreases, figure 3. Cooling rate of 0.8°C/s (1.4 °F/s) results in an elongation of ~0.5% higher, compared to the cooling rate of 2.5°C/s (4.5 °F/s).

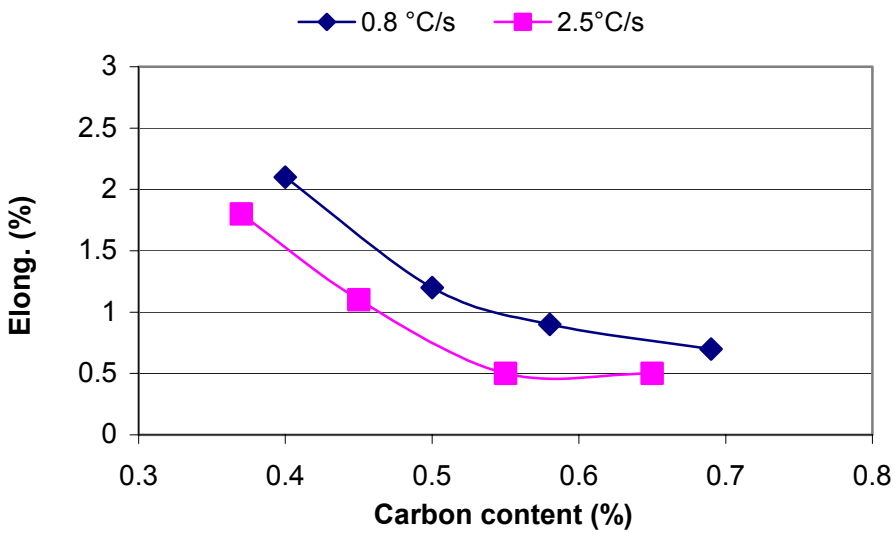


Figure 3. Elongation vs. carbon content, cooling rate 0.8 (1.4 °F/s) and 2.5°C/s (4.5°F/s).

Influence of shot peening

Shot peening operation densifies the surface to a depth range of 5 to 30µm depending on the surface hardness which is related to the material microstructure and density. No gain in yield strength was found, figure 4a. The effect from the surface densification on hardness is shown in figure 4b.

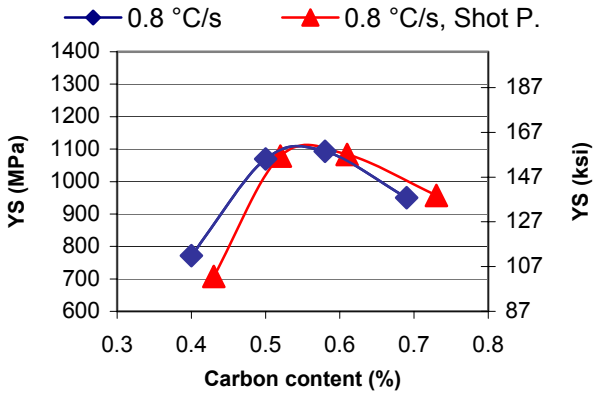


Figure 4a. Yield strength vs. of carbon content, cooling rate 0.8°C/s (1.4 °F/s).

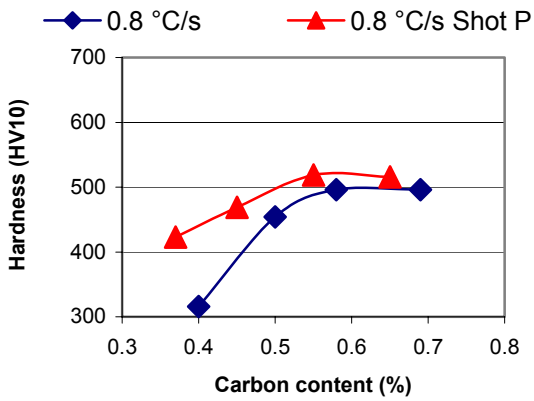


Figure 4b. Hardness, HV10, vs of carbon content, 0.8°C/s (1.4 °F/s).

Dynamic properties

Four point plane bending fatigue, R= -1 frequency 25 Hz, run out at 2 million cycles was evaluated at a cooling rate of 0.8°C/s (1.4 °F/s). Fatigue strength limit [$\sigma_{A\ 50}$] was determined with stair case method according to MPIF standard no. 56, 2001. Results obtained are summarised in figure 5 [3]. A fatigue strength of 300 MPa (43 ksi) is found at carbon content of 0.4%. By raising the carbon content to 0.5% C, an optimum is found regarding static properties, while the fatigue strength goes up to 390 MPa (57 ksi). At higher carbon content the static properties decreases but the fatigue strength continues to increase. A fatigue strength of 445 MPa (65 ksi) is found at 0.7% C. At this carbon

content the microstructure consists of martensite with some retained austenite between the martensite needles.

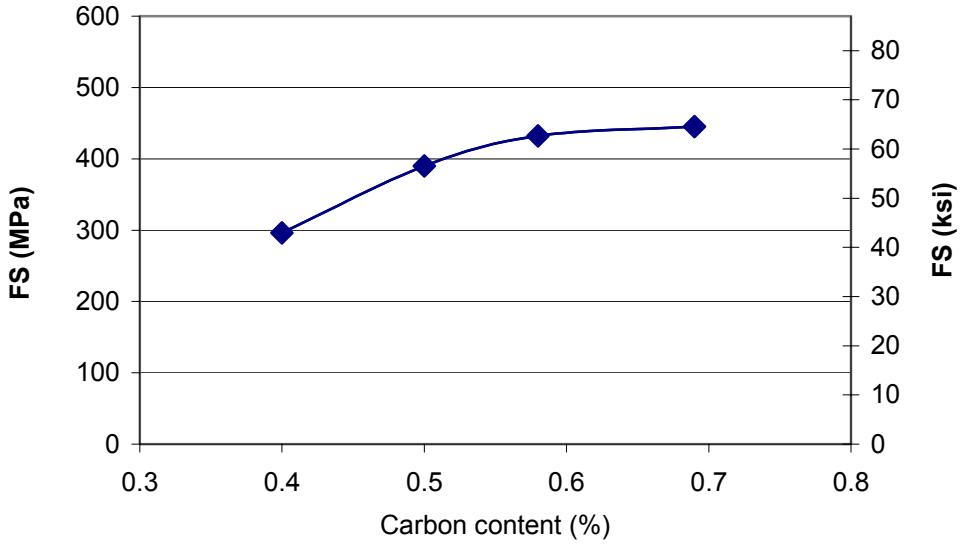


Figure 5. Fatigue strength as a function of carbon content at a cooling of 0.8°C/s (1.4 °F/s).

Fatigue strength can be increased by shot peening the samples (figure 6). At carbon content 0.4% the gain in fatigue strength is 15%. By raising the carbon content to 0.7% a fatigue strength of 520 MPa (75 ksi) is achieved correspondning to an increase of 17%.

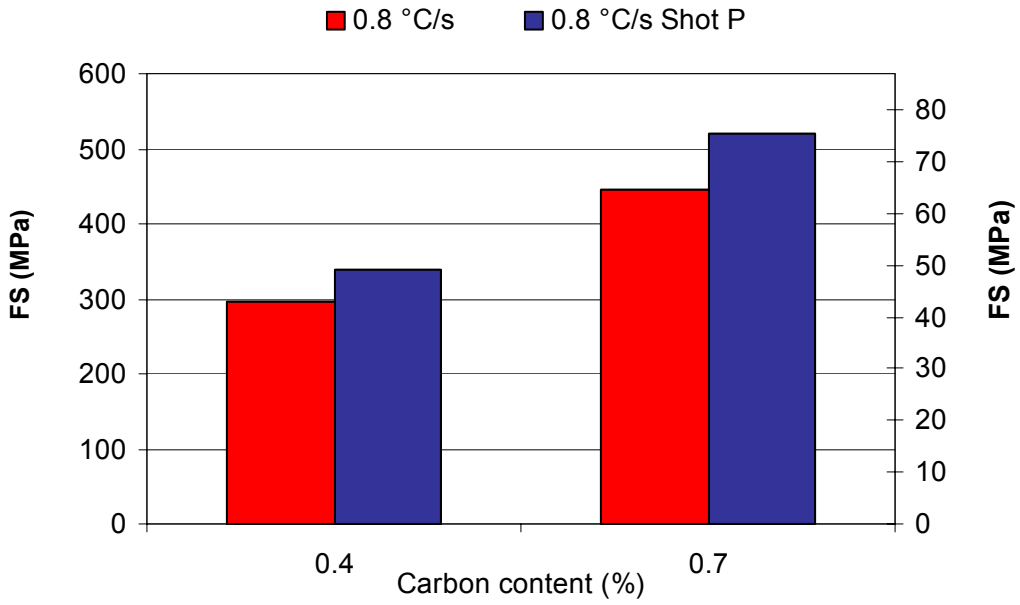


Figure 6. Influence of shot peening on fatigue strength

Heat treatment
The results obtained for material E when high temperature sintered and than vacuum carburised are summarised in table V.

Table V. Properties of vacuum carburised Astaloy CrM

Material		C as-sint %	Sint density g/cm^3	HV10	HRC	Fatigue strength MPa/ksi	
Mix No.	graphite wt%					$\sigma_{A,50\%}$	St. dev.
4	0.45	0.30	7.29	507	42	527 /76.7	<5/0.4

After processing the achieved density was 7.3 g/cm^3 and the sintered carbon content was 0.3%. The obtained hardness was 47 HRC. Fatigue strength of 527 MPa (76 ksi) was found.

Metallographic investigation carried out on the fatigue test samples showed a martensitic structure at the surface. Some retained austenite and few carbides were also observed at the surface. Mainly bainite with some martensite were found in the centre of the samples.

Microhardness HV0.1 was measured and the profile is reported in figure 7. Case hardening depth defined as the distance from the surface at which HV0.1 is below 550 Vickers units is approx. 0.4 mm.

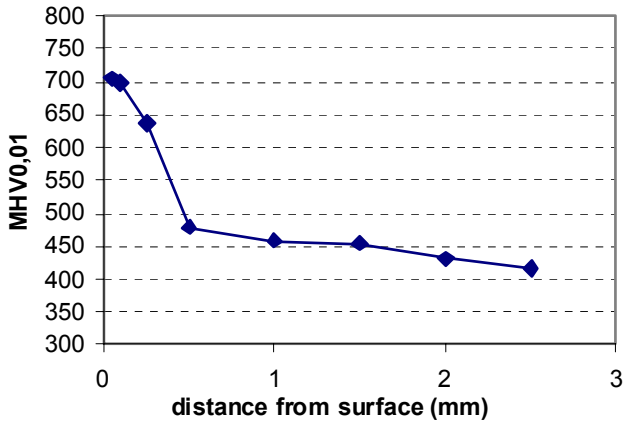


Figure 7. Microhardness profile

DISCUSSION

Raising the sintering temperature from 1120 °C (2050 °F) to over 1200 °C (2192 °F) is beneficial both for the strength and toughness of Astaloy CrM [4]. As the alloying elements chromium and molybdenum are prealloyed to the iron the reason for the higher mechanical properties obtained after high temperature sintering, if as-sintered carbon content, sintered density and cooling rate are kept constant, is the formation of more developed sintering necks between the powder particles.

Due to the presence of chromium and molybdenum Astaloy CrM has a good hardenability. An almost enterely martensitic structure (> 85% martensite) is obtained at 0.55% carbon when cooling at the rate of 0.8 °C/s (1.4 °F/s). At this carbon level and density of 7.15 g/cm^3 a hardness of 45 HRC is obtained in combination with a yield strength of 1092 MPa (158 ksi), elongation of 1.3% and bending fatigue of 410 MPa (59 ksi).

At 0.55% C a maximum in yield properties is reached. Further increase in carbon content results in decreased static properties due to increased fragility of the martensite and formation of retained austenite [5]. Dynamic properties instead increases when raising the carbon content from 0.4 to 0.7% and a fatigue strength of 445 MPa (65 ksi) is obtained at 0.7% C. This is in compliance with the behavior of D.AE and D.HP-1.

Optimum carbon content when cooling at 2.5 °C/s (4.5 °F/s) is 0.45% which is lower if compared to 0.8°C/s (1.4 °F/s) in cooling rate [5]. Increased cooling rate, 2.5°C/s (4.5 °F/s) gives an increase in yield strength by ~150 MPa (22 ksi) reaching 1245 MPa (181 ksi) at a combined carbon content of 0.45%. This at the expense of the elongation which decreases by 0.5%.

For carbon content $\geq$ 0.55% Astaloy CrM has a hardenss HRC of 45 units indipendently of the cooling rate. At 0.55% the material has an almost enterely matensitic structure both when cooling at 0.8 °C/s (1.4 °F/s) and 2.5 °C/s (4.5 °F/s). When further increasing the carbon content no further increase in hardness HRC is observed due to the formation of retained austenite.

Shot peening introduces compressive stresses in the material, if the microstructure can take plastic deformation, contributing in this way to an improvement in fatigue properties. Modification of crack initiation sites at the surface i.e closure of surface pores is another mechanism that will enhance the fatigue performance. By carrying out a shot peening operation after high temperature sintering followed by cooling at 0.8 °C/s (1.4 °F/s) an increase in fatigue strength by 15% was obtained. At 0.7% C a fatigue strength of 520 MPa (75 ksi) was reached.

Many components, gears for example, require a hard surface and a tough core. These properties are commonly achieved by case hardening operations such as carburising. In order to eliminate the risk of oxidation, the possibility of combining high temperature sintering with carburising in vacuum was investigated. The advantage of this process is that sintering and the heat treatment operation can be carried out in one step. A fatigue limit of 527 MPa was obtained at heat treatment conditions tested.

CONCLUSIONS

- High temperature sintered Astaloy CrM is suitable for manufacturing high performance PM parts.
- Plane bending fatigue strength increases from 300 MPa (43 ksi) to 445 MPa (65 ksi) when raising the carbon content from 0.4-0.7%C.
- Fatigue strength can be further increased by carrying out a shot peenening operation. A plane bending fatigue strenght of 520 MPa (75 ksi) can be achieved at 0.7%C and density 7.2 g/cm^3.
- Optimum yield strength, 1093 MPa (158 ksi), is obtained at 0.58% C when cooling at the rate of 0.8°C/s (1.8 °F/s).

- By combinig high temperature sintering with rapid cooling optimum yield strength, 1245 MPa (181 ksi), is obtained at 0.45% C.
- Hardness of 45 HRC is obtained at 7.2 g/cm^3 in density for carbon content above 0.5% C, indipendently of cooling rate.
- Warm compaction combined with high temperature sintering and vacuum carburising of Astaloy CrM with 0.3% C and a density of 7.3 g/cm^3 gives a plane bending fatigue strength of 527 MPa (76 ksi).

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