PERFORMANCE CHARACTERISTICS OF HIGH PERFORMANCE DIFFUSION BONDED MATERIALS

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ABSTRACT

It has long been known that diffusion bonding elements such as copper, nickel, and molybdenum to iron can offer excellent performance, high compressibility, and very stable dimensional performance. These characteristics are enhanced when copper and/or nickel are diffusion bonded to a 1.5% Mo prealloyed base. This paper describes the performance available from systems using 2% Ni and 4% Ni + 2% Cu diffusion bonded to a 1.5% Mo prealloyed base. Properties achieved by conventional compaction, conventional and high temperature sintering, and sinterhardening will be discussed.

INTRODUCTION

The development of new steel powders and processes during the last decades has led to a considerable expansion of the applications for PM. In order to further extend the applications for PM there is a demand for alloy systems as well as processes which result in improved mechanical properties with maintained precision of tolerance control at reduced costs. As dimensional accuracy is of prime concern, the efforts to further increase the mechanical properties of the materials automatically create a dilemma. Above a certain strength level the parts cannot be sized. There are two possible ways to overcome this problem. One is to sinter the parts to a moderate strength, then size them and finally heat treat them to the desired high strength. The drawback with this method is the dimensional change upon heat treatment which may throw the parts out of size very easily. The other possibility is to have a precise control over the whole process so the parts come out of the sintering furnace with the desired tolerances and strength.

In this paper two diffusion alloyed materials which combine high strength with a small dimensional change after normal processing conditions are presented. The paper is divided into three different parts. The first part deals with a diffusion alloyed powder that results in a small variation in dimensional change during sintering independently of the density within the component. The second part focuses on a diffusion alloyed powder containing 4%Ni and 2%Cu material that reaches very high strength already after sintering. In the final part the 2%Ni diffusion bonded material response to sinterhardening is discussed. The effect of using higher sintering temperatures than 1120°C/2050°F in combination with increased cooling rate for these materials is also presented.

PART 1- MATERIAL WITH STABLE DIMENSIONAL CHANGE

There are often internal variations in density when components are compacted to different internal height levels. Examples of such components are for instance synchronising hubs. To maintain the tolerances from the green to the sintered condition it is of interest to have a uniform dimensional change independent of the internal density variations. For this purpose a diffusion alloyed powder, D. DC-1, has been developed making it possible to produce new and more complex sintered components and still maintain good precision of tolerances [1].

POWDERS AND PROCESSING CONDITIONS

The powders used in the first part of this presentation are showed in table 1 below. D. AE is based on a high compressibility iron powder, ASC 100.29, and diffusion alloyed with 4% Ni, 1,5 % Cu and 0,5% Mo. This powder has been a work horse in the PM industry for a long time and is used as a reference throughout this paper. This material is designated FD-0400 in the MPIF nomenclature. D. DC-1 is based on a 1,5% Mo pre-alloyed powder (Astaloy Mo) diffusion bonded with 2% Ni. Both powders were mixed with 0.8 % Acrawax and 0.5 % graphite. Mixes containing 0.75 and 0.95 % graphite were also prepared with the D. DC-1 powder to study the dimensional and strength performance at higher carbon contents.

Table 1: Chemical composition of the investigated powders

Powder grade	Base powder	Material code	Cu wt %	Ni wt%	Mo wt%	Graphite wt%	A-wax wt%
D.AE	ASC 100.29	A	1.5	4	0.5	0.5	0.8
D.DC-1	Astaloy Mo	B		2	1.5	0.5-0.9	0.8

Test specimens were compacted at compacting pressures in the range of 400 to 800 Mpa (30-58tsi). Sintering was carried out in a mesh belt furnace at 1120°C (2050°F)for 30 minutes. The cooling rate was approximately 0.8°C/s (1.6°F/s) in the temperature range 800-300°C (1470-570°F). A sintering atmosphere consisting of 90/10 nitrogen/hydrogen was used with addition of methane for carbon control. Green and sintered properties were analyzed according to standardized test methods. The dimensional change was measured from the green size to the sintered size on tensile strength testbars.

RESULTS

Influence of powder grade

The density level obtained with the two materials was slightly different due to differences in compressibility. At a carbon content of 0.5%, material A shows an increasing dimensional change with increasing density, figure 1. A density variation of 0.5 g/cm^3 results in a dimensional change variation of $\pm$ 0.05 % for this material. Material B on the other hand shows an almost stable dimensional change independent of density in the density range 6.75-7.25 g/cm^3.

Measurements of the dimensions were also made on the synchronising hub shown in figure 2. The hub has an over pin diameter of 95.0 mm. In the green condition the scatter of the diameter, $\pm 3\sigma$ (σ = standard deviation), was about the same for material A and B, figure 3.

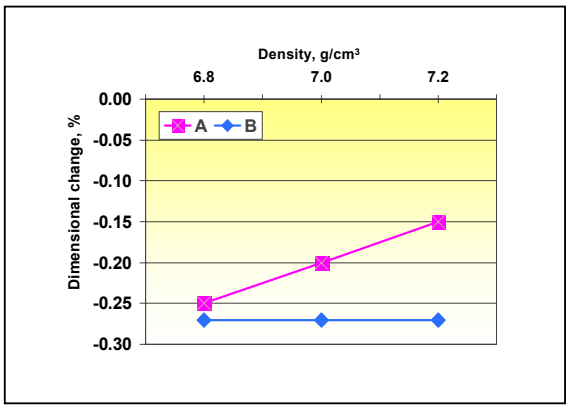


Fig. 1. Dimensional change at different densities.

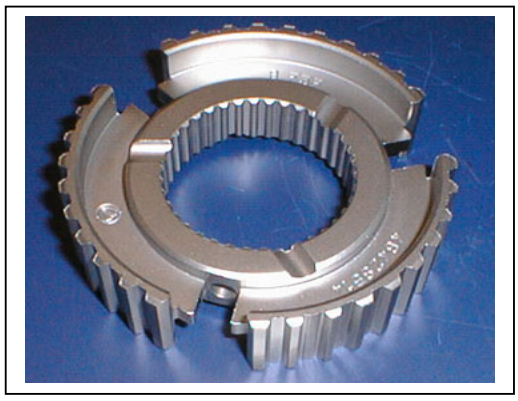


Fig. 2. Synchronising hub, a component with. internal density variations.

Sintering was performed in two production sintering furnaces under different sintering conditions. In both cases the sintering temperature used was 1120°C (2050°F) but sintering time and cooling rate were different. In one furnace the sintering time was 40 minutes and a cooling rate of 0.5°C/s (1.0°F/s) was used. In the other furnace a sintering time of 112 minutes and a cooling rate of 0.25°C/s (0.5°F/s) were used. As can be seen from the results in figure 3 material B showed a smaller scatter of the sintered diameter for both sintering conditions.

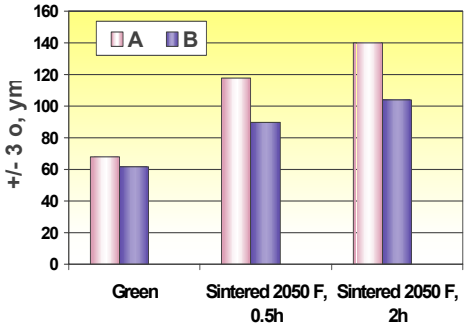


Fig. 3. Scatter in outer diameter of synchronising hub

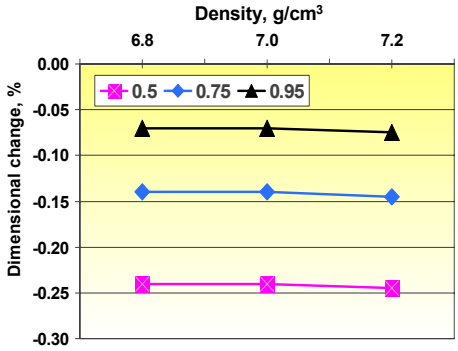


Fig. 4. Dimensional change at different carbon contents of material B.

The influence of different added amount of graphite on the dimensional change was also studied for material B. The added amounts of graphite were 0.5, 0.75 and 0.95 % respectively. As can be seen from figure 4 the dimensional change shows to be independent of density for all three carbon contents even though the different carbon contents result in different dimensional change levels.

Sintered properties

Mechanical properties obtained after compaction at 600 MPa (43tsi) and sintering at 1120°C (2050°F) are shown in table 2 below. The tensile strength is about 30 MPa higher for material A, whereas the yield strength is 140 MPa higher for material B. The latter is often of greater interest as hardly any plastic

deformation can be accepted for PM components. The higher yield strength of material B is due to the pre-alloying of Mo in the base powder. Hardness and elongation are slightly higher for material A.

Table 2. Mechanical properties for material A and B after compaction at 600 MPa /43tsi and sintering at 1120°C/2050F for 30 min in 90/10 nitrogen /hydrogen atmosphere

Material	C %	SD g/cm3	HV10	TS MPa (ksi)	YS MPa (ksi)	A %
A	0.49	7.00	200	721	362	3.1
B	0.49	7.00	184	690	502	2.5

From figure 5 it can be seen that the tensile strength of both materials increase almost linear with increasing green density. The tensile strength is increased about 30% for both materials when the density is increased from 6.8 to 7.2 g/cm3. By increasing the carbon content of material B from 0.5 to 0.75 % the tensile strength is increased about 50 Mpa at all density levels, figure 6. At the highest carbon content, 0.95 %, the strength is decreased.

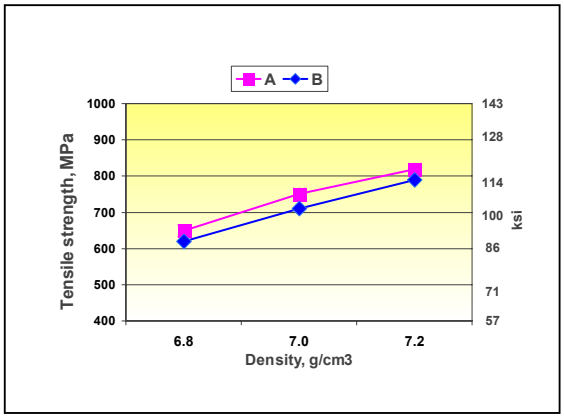


Fig. 5. Tensile strength at different densities of material A and B

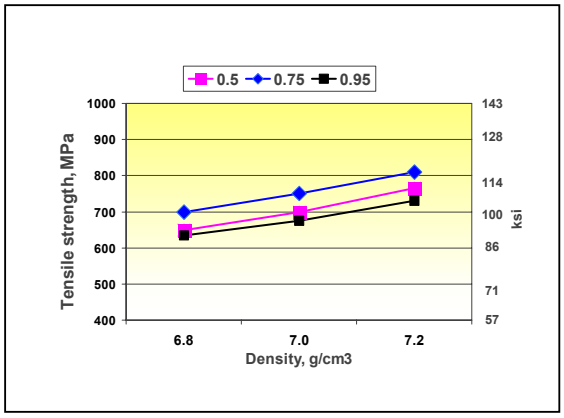


Fig. 6. Tensile strength at different carbon contents and densities of material B.

The microstructure of material B with 0.5 % C consists of both upper and lower (coarse and fine) bainite, some martensite and retained austenite. With 0.75 % C the bainite is finer due to the lower transformation temperature and the amount is decreased whereas the amount of retained austenite has increased. The drop in strength at the highest carbon content, 0.95 %, is due to the formation of grain boundary cementite.

Summary part 1

The optimized chemical composition and the alloying technique of D. DC-1 results in even dimensional change independent of density variations within the component. The dimensional change is also independent of density variations at different carbon levels. The material has a high yield strength of 500 MPa at an addition of 0.5 % carbon. The corresponding tensile strength is 700 MPa. The latter is in the same range as for D. AE. The strength properties are improved for increasing carbon contents up to 0.75 %. Higher contents can result in grain boundary cementite.

PART 2 – HIGH STRENGTH MATERIALS

In the second part of this paper the two previously described powders D. AE and D. DC-1 are compared with another diffusion alloyed material, D. HP-1. Like D. DC-1 this powder is also based on the 1,5% Mo pre-alloyed powder Astaloy Mo but diffusion bonded with 4% Ni and 2% Cu [2]. The chemical composition of the powder mixes studied are reported in table 3 below.

Table 3: Chemical composition of the investigated materials

Powder Grade	Material code	Cu wt %	Ni wt%	Mo wt%	Graphite wt%	A-wax wt%
D. AE	A	1.5	4	0.5	0.6	0.8
D. DC-1	B		2	1.5	0.6	0.8
D. HP-1	C	2	4	1.5	0.6	0.8

Test specimens were compacted at compacting pressures in the range of 400 to 800 Mpa (30-58 tsi). Sintering was carried out using two different conditions. Sintering at 1120°C (2050°F) was carried out in a mesh belt furnace. The cooling rate was approximately 0.5°C/s (1.0°F/s) in the temperature range 800-300°C (1470-570°F). An atmosphere consisting of 90/10 N2/H2 was used with addition of methane for carbon control. Sintering was also carried out in a high temperature furnace at 1250°C (2282°F) for 30 minutes in a nitrogen / hydrogen atmosphere. The cooling rate was approximately the same as for thesintering carried out at 1120°C (2050°F).

Green and sintered properties were analysed according to standardised test methods. The dimensional change was measured from the green size to the sintered size on tensile strength testbars. Fatigue testing was carried out with four point bending using as-sintered ISO fatigue test bars. Test mode was R= -1.

RESULTS

Sintered properties at 1120°C (2050°F)

From the tensile strength figures in table 4 it can be seen that the tensile strength levels obtained at a carbon content of 0.5 % are much higher for material C (D.HP-1) than for material A and B when sintered at 1120°C (2050°F) for 30 minutes. The tensile strength for material C is more than 20% higher than for material A and 25% higher than for material B. The hardness level obtained for material C is 20-30 units higher than for the other two materials. The elongation is slightly lower for material B and C compared to material A. However, the elongation obtained for material C is still high for such a hard material. All materials shrink slightly during sintering, about -0.15 %. The sintered density for material B and C is slightly lower compared to material A. This is a consequence of their slightly lower shrinkage but also an effect of the slightly lower compressibility of powder of B and C.

Table 4. Properties of material A, B and C after sintering at 1120°C (2050°F) for 30 minutes. Compacting pressure 600 MPa (43 tsi), Carbon content 0.5 %.

Material code	Powder	Density g/cc	Dim. Change %	Tensile Strength MPa (ksi)	Elongation %	Hardness HV10
A	D. AE	7.11	-0.18	739	2.9	210
B	D.DC-1	7.07	-0.12	715	2.3	201
C	D.HP-1	7.07	-0.15	893	2.3	236

In figure 7 the influence of density on tensile strength of the three different materials is shown. It can be seen that the tensile strength is increased about 200 MPa for all materials when the density is increased from 6.8 to 7.2 g/cm^3. Material C obtains about 200 MPa higher tensile strength than material A and B at all density levels. At a density of 7.2 g/cm^3 a tensile strength of more than 1000 MPa is possible to achieve with material C.

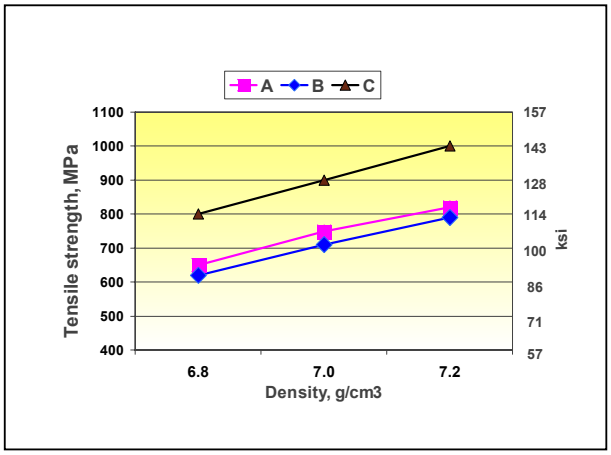


Fig. 7. Tensile strength of material A, B and C after different densites after sintering at 1120°C (2050°F) for 30 minutes

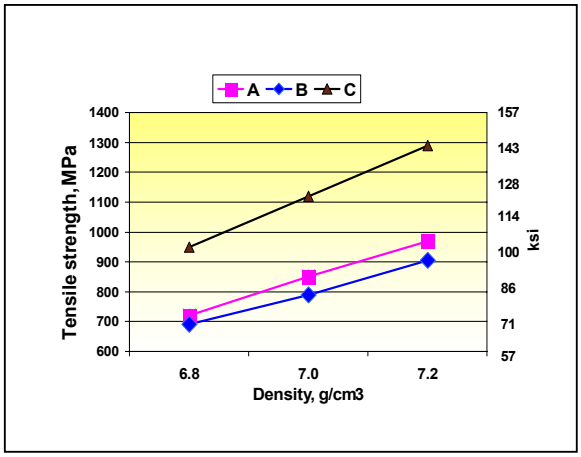


Fig. 8. Tensile strength of material A, B and C at different densites after sintering at1250°C(2280°F) for 30 minutes

Sintered properties at 1250°C (2282°F)

By increasing the sintering temperature to 1250°C (2282°F) the tensile strength can be increased substantially for all materials and specifically for material C. As can be seen from table 5 a tensile strength of 1174 MPa was obtained already after sintering for this material. This is almost 50 % higher than the tensile strength obtained with material A and B. Material A obtained slightly higher tensile strength level compared to material B. At the higher sintering temperature the tensile strength increased about 15% for these two materials.

Hardness also increased for all materials. Material C achieved a hardness of 326 compared to about 250 for material A and B. The highest elongation was obtained for material A and slightly lower for material B. Material C obtained 1.5% which is still good considdering the high tensile strength and hardness level. The dimensional change during sintering showed an increased shrinkage for all three materials, -0.52% to -0.59 %. The increased shrinkage contributes to a slight density increse for all materials compared to the densities obtained at the lower sintering temperature.

Table 5. Properties of material A, B and C after sintering at 1250°C (2280°F) for 30 minutes. Compacting pressure 600 MPa (43 tsi), Carbon content 0.5 %.

Material code	Powder grade	Density g/cc	Dim. Change %	Tensile Strength MPa (ksi)	Elongation %	Hardness HV10
A	D. AE	7.13	-0.55	830	2.5	250
B	D.DC-1	7.09	-0.52	810	2.3	245
C	D.HP-1	7.12	-0.59	1174	1.5	326

In figure 8 the influence of density on tensile strength of the different materials is shown. The tensile strength increased about 200 MPa for all materials when the density is increased from 6.8 to 7.2 g/cm^3. Material A achieved a slightly higher strength than material B at all densitiy levels. Material C obtained a substantially higher tensile strength compared to the other two materilas. As can be seen the tensile strength is 200-300 MPa higher at the different density levels. At a density of 7.2 g/cm^3 a tensile strength of more than 1300 MPa is possible to achieve with material C.

Fatigue strength

The bending fatigue strength obtained for the three materials at different carbon contents are shown in figure 9 [3]. As can be seen the fatigue strength is increased with increased carbon content for all materials. Material C shows the largest increase while material B shows the smallest. The highest fatigue strength at all carbon levels was obtained with material C. This material has about 30 to 50 MPa higher fatigue strength than material A. Material B shows comparable fatigue strength to material A at the low er carbon level but is about 40 MPa lower at the higher carbon contents. The highest fatigue strength is obtained with material C at a carbon content of 0.85%. At this carbon level a fatigue strength of 330 MPa was achieved.

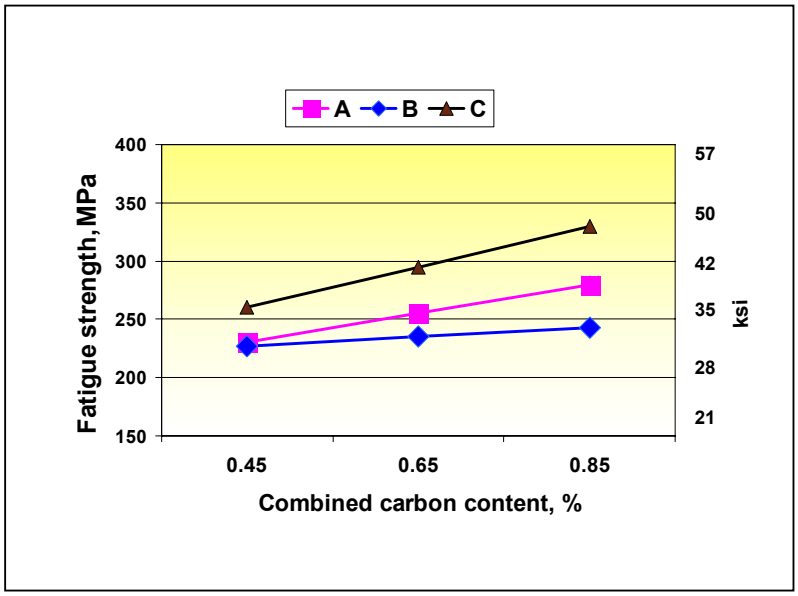
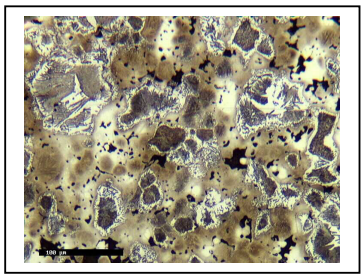


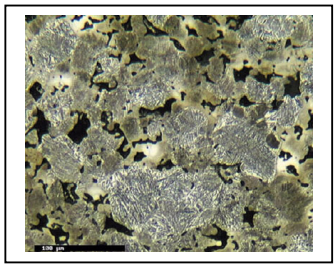
Fig 9. Bending fatigue strength of material A, B and C at different carbon contents after sintering at and 1120°C(2050°F) for 30 minutes. Density 7.10g/cm3

Microstructures

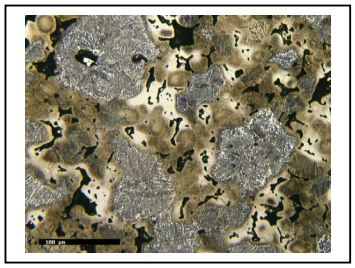
The microstructures obtained for the three different materials at 0.5% carbon content are shown in figure 10A-C after sintering at the 1120°C (2050°F) and in figure 11A-C for the materials sintered at 1250°C (2280°F) [4]. As can be seen all materials have a heterogeneous microstructure containing pearlite, bainite, martensite and nickel rich austenite and ferrite in the case of material A after sintering at the lower temperature. After sintering at the higher tempeature the microstructure is more homogenous for all materials. The nickel rich austenite areas can be seen in all materials. The pore rounding effect obtained at the higher sintering temperature can be clearly seen in figure 11B.



10A: Material A

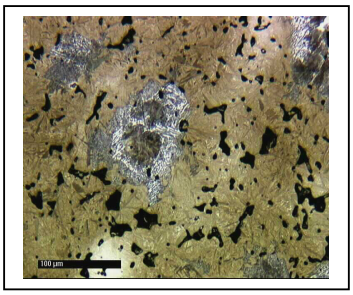


10B : Material B

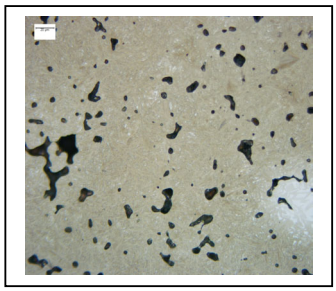


10C: Material C

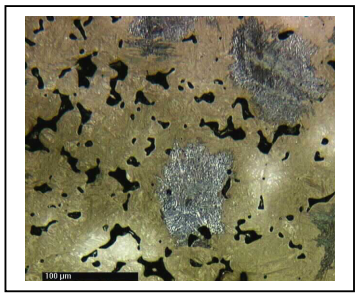
Fig 10A-C. Microstructures of for material A, B and C after sintering at 1120°C (2050°F) for 30 minutes.



11A: Material A



11B: Material B



11C: Material C

Fig 11A-C. Microstructures of for material A, B and C after sintering at 1250°C (2282°F) for 30 minutes.

Summury of part 2

By diffusion alloying Astaloy Mo with 4%Ni and 2%Cu a material that achieves a tensile strength of more than 900 MPa already after sintering at 1120°C (2050°F) is obtained. At this sintering temperature the dimensional change is close to zero which means that it is possible to maintain close tolerances. The mechanical properties of this material can be further improved by increasing sintering temperature, sintering time as well as cooling rate. By increasing the sintering temperature to 1250°C (2282°F) tensile strength levels of 1100 -1300 MPa are possible to obtain already after sintering. This is comparable to what is possible to achieve by heat treatment of materials made from D. AE.

PART 3 -EFFECT OF SINTER-HARDENING

The amount of alloying elements, alloying method, density, sintering temperature and the post-sintering cooling rate will all influence the microstructure of the material. Copper, nickel and molybdenum all increase the hardenability of the material. By accelerating the cooling rate the quantity of martensite is increased resulting in increased strength and hardness. Rising the sintering temperature from 1120°C (2048°F) to 1250°C (2282°F) results in higher densities, rounder pores as well as larger inter pore spacing. Increased sintering temperature also leads to a more even distribution of alloying elements and as a consequence increased hardenability of the base material. By combining high temperature sintering with an accelerated post-sintering cooling rate the performance of the materials can be further improved.

In this third section sinter-hardening of the diffusion alloyed material, D. DC-1, will be compared to the frequently used prealloyed PM material FL-4600 with 2 % Cu added. The influence of density, sintering temperature and cooling rate on both microstructure and mechanical properties will be discussed.

CCT-DIAGRAM

In order to study the influence of different cooling rates on the microstructure CCT-diagrams are a useful tool. The CCT diagram for the D. DC-1 material is shown in figure 12 below. Examination of the microstructure obtained of the different cooled samples revealed that independently of the cooling rate all samples contained about 2 % of retained austenite probably more dependent upon sintering time and temperature than on cooling rate. Transformation from austenite to bainite or martensite is however strongly influenced by the cooling rate which can be seen in figure 13. The amount of martensite increases from about 10 % in the sample cooled at 0.25°C/s up to 98 % at 70°C/s. The bainitic structure becomes finer at higher temperature gradients.

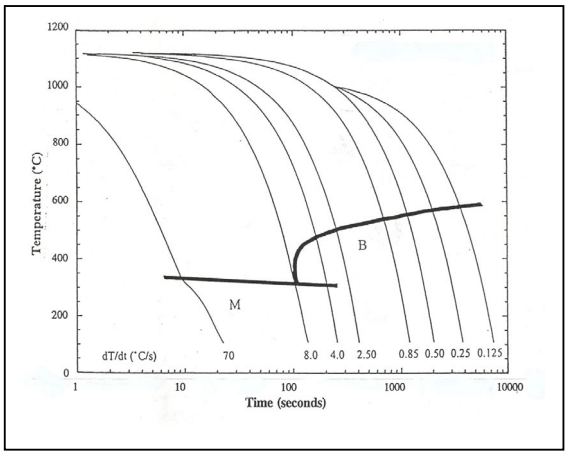


Fig. 12 CCT-diagram for DC+0.7%C

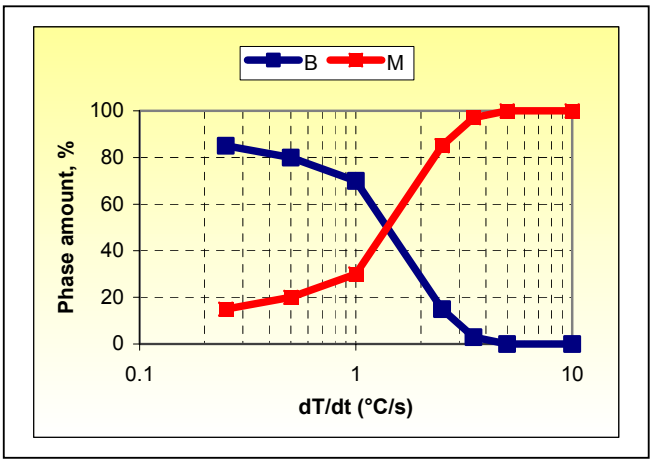


Fig. 13 Phase amount vs cooling rate for DC material

MATERIALS AND PROCESSING CONDITIONS

The influence of increased cooling rate on the mechanical properties and hardness was studied for the diffusion alloyed material D. DC-1. The prealloyed PM material FL4600 with 2% Cu added was used for comparison as this is a frequently used material for sinter hardening applications. 0.8 % graphite was admixed to both powders. Tensile-, impact energy- and transverse rupture strength samples were pressed to a green density of 6.9 g/cm^3. Two sintering conditions were used, as summarised in table 6. Sintering at 1120°C (2048°F) was carried in a laboratory mesh belt furnaces equipped with a rapid cooling unit. Sintering at 1250°C (2282°F) was carried out in a lifting earth furnace equipped with a unit for rapid cooling. All materials were tempered at 200°C (390°F) for 60 minutes in air. Cooling rate was determined by comparing the microstructure of the investigated materials with the CCT diagram described above [5].

Table 6: Sintering conditions

	Test 1	Test 2	Test 3	Test 4
Temperature	1120 °C/2048 °F	1120 °C/2048 °F	1250 °C/ 2282 °F	1250°C/ 2282 °F
Time	30 min	30 min	30 min	30 min
Cooling rate	0.25-0.8 °C/s 0.45-1.4 °F/s	2-3 °C/s 3.6-5.4 °F/s	0.25-0.8 °C/s 0.45-1.4°F/s	2-3 °C/s 3.6-5.4 °F/s
Atmosphere	90-10 N_2-H_2+CH_4	90-10 N_2-H_2	90-10 N_2-H_2	90-10 N_2-H_2

RESULTS

Hardness obtained after sintering at both temperatures and cooling rates are reported in figure 14. All hardness values are reported in HRC. It is clear that an increase of the cooling rate from 0.25-0.8°C/s (0.5-1.6°F/s) to 2-3°C/s (4-6°F/s) results in an excellent improvement of the hardness. When sintering at 1120°C (2050°F), higher hardness is observed for the FLC4608 material at both cooling rates. When sintering at 1250°C (2282°F), the hardness decreases slightly for this material but increases slightly for the DC material at the lower cooling rate. Substantially higher hardness is achieved with both materials by combining high temperature sintering with increased cooling rate. At the higher sintering temperature and cooling rate the DC material obtained the highest hardness, 40 HRC**.** The reason for this can be found in the microstructure described below.

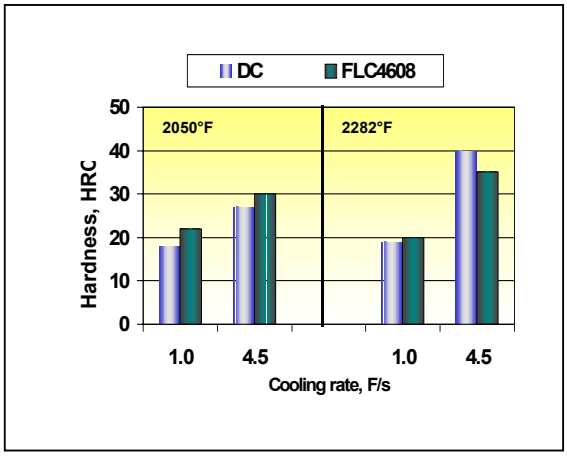


Figure 14. Hardness, HRC, after sintering at 1120°C (2050°F) and 1250°C (2282°F) with cooling rates of 0.5°C/s (1.0°F/s) and 2.5°C/s (4.5°F/s)

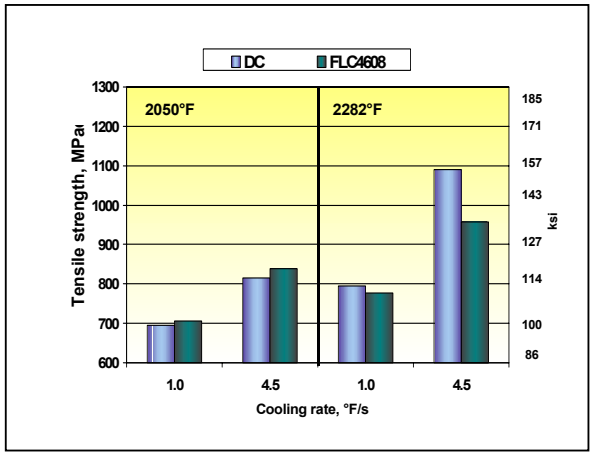


Figure 15. Tensile strength after sintering at 1120°C (2050°F) and 1250°C (2282°F) with cooling rates of 0.5°C/s (1.0°F/s) and 2.5°C/s (4.5°F/s)

From figure 15 it can be seen that tensile strength increases with rising sintering temperature for both materials. At the lower sintering temperature the FLC 4608 material achieved slightly higher tensile strength at both cooling rates compared to the DC matreial. However at the higher sintering temperature tensile strength is higher for the DC. Especially at the higher cooling rate the tensile strength is increased substantially for the DC material. As can be seen a tensile strength of about 1100 MPa is obtained for this material compared to 950 MPa for the FLC 4608 material. The improvements of the tensile strength is mainly explained by the increasing amount of martensite obtained for the DC material when high temperature sintered. Some improvements are also related to better sinter necks and pore roundness and the fact that this material shows the largest improvement in sintered density.

The transverse rupture strengths obtained shows very similar behaviour as the tensile strength, see figure 16. At the lower sintering temperature the transverse rupture strength is comparable for both materials at both cooling rates. The FLC 4608 material shows slightly higher transverse rupture strength. However at the higher sintering temperature the transverse rupture strength is higher for the DC material, especially in combination with the higher cooling rate.

The impact energy results obtained are shown in figure 17. At the lower sintering temperature the impact energy is comparable for both materials at both cooling rates. At the higher sintering temperature the

impact energy increased for both materials and the highest impact energy was obtained at the higher cooling rate. For the DC material impact energy increased about 100% to 25 J at both cooling rates.

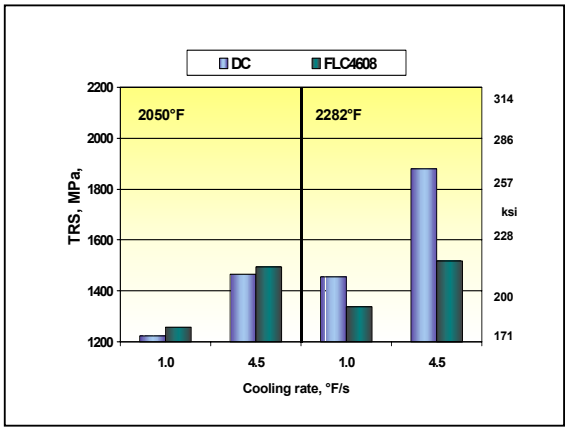


Figure 16. Transverse rupture strength after at Sintering 1120°C (2050°F) and 1250°C (2282°F) with cooling rates of 0.5°C/s (1.0°F/s) and 2.5°C/s (4.5°F/s)

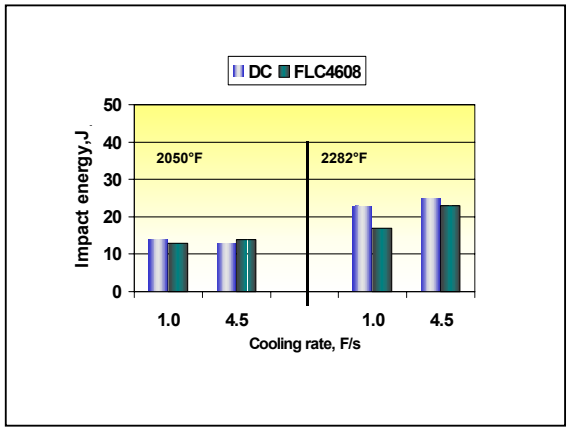


Figure 17. Impact energy after sintering at 1120°C (2050°F) and 1250°C (2282°F) with cooling rates of 0.5°C/s (1.0°F/s) and 2.5°C/s (4.5°F/s)

Microstructure

Table 9 shows the phase analysis of the microstructures obtained. FLC 4608 forms almost 100% martensite at both sintering temperatures whereas for the DC material the quantity of martensite increases with increasing sintering temperature. Some retained austenite was observed between the martensite needles in the FLC 4608 material with copper addition. The higher sintering temperature also allow for better diffusion of alloying elements and therefore results in a better hardenability of the matrix.

Table 9: Phase analysis for cooling rate 2-3°C/s (4-6°F/s)

Material	Sintering temperature	C %	Martensite %	Martensite + austenite %	Bainite %
FLC 4608	1120°C (2050°F)	0.72	Bal.		1-2%
DC		0.73		Bal.	15-25%
FLC 4608	1250°C (2282°F)	0.70	Bal.		<1%
DC		0.73		Bal.	1-2%

Summury of part 3

The properties of D.DC-1 are comparable to the properties of FLC4608 when sintered at 1120 C (2050F) at both cooling rates. The properties can be further improved by increased sintering temperature. By combining high temperature sintering at 1250°C (2282°F) with a cooling rate of 2-3°C/s (4-6°F/s) a hardness level of HRC 40 and tensile strength level of 1100 MPa are possible to obtain already after sintering with the D. DC-1 material. An impact energy of 26 J is obtained at these processing conditions.

CONCLUSIONS

The two diffusion bonded materials D.DC-1 and D.HP-1 described in this paper offer means to deliver high strength, high precision tolerances in combination with a robustness during manufacturing. This robustness, and the fact that high performance can be gained from a single operation, makes it possible to achieve the right performance levels at a low total cost.

D.DC-1 (Dimensional Control) is a material that offers an important benefit for many applications as the dimensional change during sintering remains the same, irrespective of density within the component.

D.HP-1 (High Performance) provides very high tensile and fatigue strength in the as sintered condition that are increasingly in demand for enhancing component performance. By increasing sintering temperature to 1250°C (2282°F) tensile strength levels of 1100 -1300 MPa are possible to obtain already after sintering.

By combining high temperature sintering at 1250°C (2282°F) with a cooling rate of 2-3°C/s (4-6°F/s) a hardness level of HRC 40 and tensile strength level of 1100 MPa are possible to obtain with D. DC-1 already after sintering. This is comparable to what is possible to achieve by heat treatment of materials made from D. AE.

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