DESIGNING LOW ALLOY STEEL POWDERS FOR SINTERHARDENING APPLICATIONS

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ABSTRACT

Sinterhardening is an attractive technique for the manufacturing of high hardness P/M parts because it eliminates the need for post sintering heat treatment, thus significantly reducing processing costs. Furthermore, high thermal stresses and part distortion resulting from conventional quenching are avoided, providing improved control on final part dimensions.

In order to evaluate the effect of alloying elements on sinterhardenability of different materials, a test matrix was designed to conduct comparative evaluation of various combinations of molybdenum, nickel, manganese and chromium concentrations in water atomized steel powders. Following atomization and downstream processing of experimental steel powders, these were admixed with graphite, copper and lubricant, pressed to 6.8 g/cm^3 and sintered at 1120°C and tempered 1 hour at 205°C. Additions of manganese and chromium were found to improve the hardenability of low alloy steel powders.

INTRODUCTION

Sinterhardening can be defined as a process where P/M parts transform partially or completely into a martensitic structure during the cooling phase of the sintering cycle. In order to achieve optimum hardenability, prealloying techniques are generally preferred to elemental additions. Manganese is added to all commercial steels in the range of 0.25 to 1.0% to increase strength and hardenability of plain carbon steels. Chromium is also a very popular element to improve hardenability, strength and wear resistance of conventional steels. However, in P/M steels, manganese and chromium contents are generally maintained below 0.3% because of the difficulty to reduce their oxides during annealing [1]. Molybdenum and nickel are commonly used in low alloy P/M steel powders because their oxides are easily reduced during the annealing treatment of water atomized powders. Molybdenum is very efficient for increasing the strength and hardenability of steels while nickel increases the hardenability, strength, toughness and fatigue resistance[2]. These elements are however more expensive than manganese or chromium and are subject to large market price variations.

An R&D program was carried out to develop a new prealloy steel powder with improved hardenability to promote sinterhardening in conventional sintering furnaces. In order to reduce the development time and the production scale-up of this new powder grade, the program was initiated using the Advanced Product Quality Planning principles[3].

The specific objective of the program was to develop a steel powder capable of achieving a minimum apparent hardness of 30 HRC after sintering in conventional furnaces, while maintaining powder compressibility above 6.8 g/cm^3 at 40 tsi (550 MPa).

EXPERIMENTAL PROCEDURE

Alloying elements can be used in different combinations to increase hardenability of steels. Figure 1 illustrates the effect of molybdenum, manganese, nickel and chromium concentrations on the hardenability multiplying factor [4]. Manganese has the most pronounced effect on hardenability followed by the molybdenum, chromium and nickel. However, since molybdenum and nickel are expensive alloying elements, it was decided to substitute a certain quantity with manganese and chromium. From the Design Failure Mode and Effect Analysis (DFMEA), it was established that manganese and chromium were critical elements because of their tendency to oxidize and, hence, the risk of deteriorating the compressibility and the sintered properties.

In order to quantify the effects of alloying elements on properties of P/M steels, a series of experimental powders were prepared in the QMP process development laboratory using a 200 kg capacity induction furnace. High purity steel was remelted with ferromanganese, ferrochromium, ferromolybdenum and nickel to achieve the desired steel chemistry. Table 1 shows the test matrix used in the program.

After water atomization, the powder was dried, screened, annealed and the sintered cake was pulverized and homogenized in a blender prior to the evaluation. The various powders were analyzed for chemical composition and blended with 0.8% graphite, 2% copper and 0.75% zinc stearate. Test specimens were pressed to 6.8 g/cm^3 and sintered 25 minutes at 1120°C in a nitrogen/hydrogen atmosphere in a ratio of 90/10 and tempered one hour in air at 205°C. Transverse rupture strength was evaluated according to MPIF standard 41 while tensile properties were determined using round machined specimens according to MPIF standard 10. Finally, impact energy was measured according to MPIF standard 40.

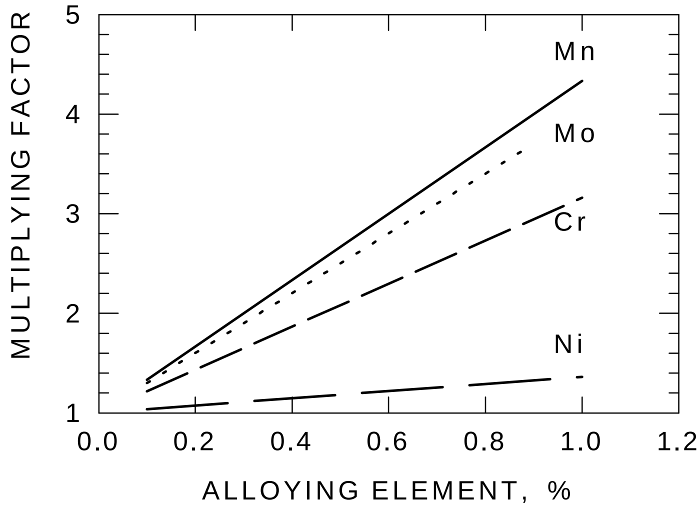

Figure 1. Hardenability multiplying factor of manganese, molybdenum, chromium and nickel [4].

TABLE 1
Targeted chemical concentrations for each experimental trial.

	Mn, %	Mo, %	Ni, %	Cr,%	Hardenability factor
1	0.40	1.25	1.9	0.05	21.5
2	0.40	0.70	1.1	0.05	11.4
3	0.85	1.25	1.1	0.05	29.0
4	0.85	0.70	1.9	0.05	23.4
5	0.70	0.60	1.0	0.60	29.7

Additional tests were performed on disc specimens weighing 450 to 1345g to evaluate the effect of the specimen size on the apparent hardness and the microstructure. For this portion of the study, mixes containing 1.0% graphite, 2% copper and 0.75% zinc stearate were prepared from trials 1, 3, 4 and 5. These were pressed to 6.8 g/cm^3, sintered 20 minutes at 1120°C in an industrial sintering furnace using a cooling rate of 0.75°C/s in the range of 870 to 650°C. The effect of a faster cooling rate, 1.5°C/s, was also studied for material #5.

RESULTS AND DISCUSSION

The chemical, green and sintered properties of the different experimental alloys are given in Table 2. The analysis of the mean effect of manganese, molybdenum and nickel (trials 1 to 4) was done using Taguchi techniques. Results of the fifth trial, with prealloyed chromium, was also added on the different figures to illustrate its effect on the various properties.

TABLE 2
Chemical, green and sintered properties of the different alloys.

	Mn %	Mo %	Ni %	Cr %	O %	Comp. Press. tsi (MPa)	Hard. (sint.) HRC	TRS kpsi (MPa)	UTS kpsi (MPa)	YS kpsi (MPa)	Elong. %	I.E. ft.lb (J)	Hard. (temp.) HRC
1	0.40	1.32	1.90	0.05	0.08	38 (524)	40	211.5 (1459)	127.4 (879)	103.9 (717)	0.5	9.1 (12.3)	33
2	0.40	0.68	1.22	0.05	0.11	31 (428)	33	206.4 (1423)	102.8 (709)	83.1 (573)	0.6	6.0 (8.1)	27
3	0.86	1.23	1.12	0.05	0.17	42 (579)	41	169.0 (1166)	120.3 (830)	109.4 (754)	0.3	8.6 (11.7)	33
4	0.87	0.76	1.96	0.05	0.19	38 (524)	37	187.6 (1294)	104.9 (723)	92.1 (635)	0.4	7.0 (9.5)	30
5	0.73	0.59	1.12	0.60	0.23	42 (579)	39	217.5 (1500)	123.8 (854)	104.1 (718)	0.4	8.9 (12.1)	32

The mean effect of prealloyed manganese, molybdenum, nickel and chromium on oxygen content, apparent hardness and transverse rupture strength is illustrated in Figure 2. Oxygen content of less than 0.11% can be achieved with manganese content of 0.4%. As expected, oxygen content increases with manganese and chromium contents. Raising manganese from 0.40 to 0.86% results in an increase of the oxygen content from 0.10% to 0.18%. Prealloying P/M steel with 0.6% chromium and 0.73% manganese results in a further increase of the oxygen content to 0.23%.

It is worth noting that alloys with high hardenability factors (1, 3, 4 and 5) exhibit compacting pressures in the range of 38 to 42 tsi (524 to 579 MPa) at 6.8 g/cm^3. This was related to either high concentrations of oxygen or carbon in the annealed powders. The oxygen content is related to the quantity of manganese and chromium in the melt, hence, carbon content in the melt has to be adjusted to allow the reduction of oxygen during annealing without forming stable carbides with molybdenum and chromium.

Raising manganese content from 0.40 to 0.86% adversely affects the transverse rupture strength, a mean loss of 30,700 psi (212 MPa). The addition of 0.60% prealloyed chromium has a beneficial effect on the transverse rupture strength, a gain of about 24,000 psi (166 MPa). Increasing molybdenum from 0.72 to 1.28% and nickel from 1.17 to 1.93% has only a minor effect on transverse rupture strength.

As-sintered apparent hardness is particularly affected by molybdenum. Raising molybdenum content from 0.72 to 1.28% results in a mean gain of 5 HRC, from 35 to 40 HRC. Increasing manganese content from 0.40 to 0.86% results in a mean gain of 2.5 HRC and adding 0.60% chromium also has a beneficial effect on apparent hardness. Nickel has only a minor effect on apparent hardness. Tempering for one hour at 205°C reduces apparent hardness by an average of 7 HRC from 37.8 to 30.8 HRC but does not alter the mean effect of each alloying element.

The mean effect of prealloyed manganese, molybdenum, nickel and chromium on ultimate tensile and yield strengths, elongation and impact energy is illustrated in Figure 3. Ultimate tensile and yield strengths are mainly influenced by molybdenum and chromium. Increasing molybdenum from 0.72 to 1.28% results in a gain of about 20 kpsi (138 MPa) for both yield and ultimate tensile strengths while the addition of 0.60% chromium also improves these properties by nearly the same order of magnitude. Increasing nickel in the range of 1.17 to 1.93% and manganese in the range of 0.40 to 0.86% have only minor effects on ultimate tensile and yield strengths.

Manganese, molybdenum, nickel and chromium have no significant effect on elongation in the concentrations range of this study. Elongation of less than 1% was obtained for all of the alloys. Raising molybdenum content from 0.72 to 1.28% increases the impact energy by 2.4 ft-lb (3.3 J). The addition of chromium also has a beneficial effect on impact energy while raising nickel content from 1.17 to 1.93% results in a gain of less than 1 ft-lb (1.4 J). Raising manganese content from 0.40 to 86% has only a minor effect on impact energy.

Figure 4 presents the as-sintered apparent hardness values measured of top and bottom faces of disc specimens weighing 450, 895 and 1345g. These disc specimens were made from alloys #1, #3, #4 and #5 respectively. Results were compared to a FLC4608 alloy processed in the same sintering conditions. Disc specimens made from alloy #5 (5F) were also sintered in a furnace equipped with a fast cooling unit.

It is first seen from Figure 4 that for the small disc specimens (450g), the different alloys respond in a similar manner to sinterhardening with the exception of alloy 5F submitted to the fast cool treatment which shows the highest apparent hardness. However, the apparent hardness decreases with the increasing weight of the specimens, and the effect is more pronounced for the FLC4608 alloy. The same pattern can be observed on the cross section of the disc specimens as shown in Figure 5. This figure illustrates the apparent hardness measured on the cross section of disc specimens weighing 450, 895 and 1345 g of alloys 5 and FLC4608. The highest hardness is reached for the small disc specimens and as the weight is increased, the FLC4608 alloy shows a sharp drop of the apparent hardness. This is explained by the lower hardenability of this alloy. This is confirmed by the microstructure as illustrated in Figure 6. For the 450g disc specimens, the microstructure of the different alloys is composed of martensite and bainite with the fast cooled alloy #5 having the largest amount of martensite. For the 1345g specimens, the amount of martensite is reduced and the degree of reduction is more pronounced in alloys with lower hardenability factor, FLC4608 and alloy #1. This explains the lower apparent hardness measured on the 1345g specimens made from the FLC4608 alloy as compared to the other steels.

After completion of this first phase of the development program, optimization of the alloy chemistry was initiated with the objective of maintaining a good hardenability and improving the compressibility. In the latter case, reducing the oxygen content of the annealed powder was the key parameter. From the results of the first phase of the development, the team agreed to adjust manganese and chromium contents in the range of 0.4 to 0.5% in order to reduce the

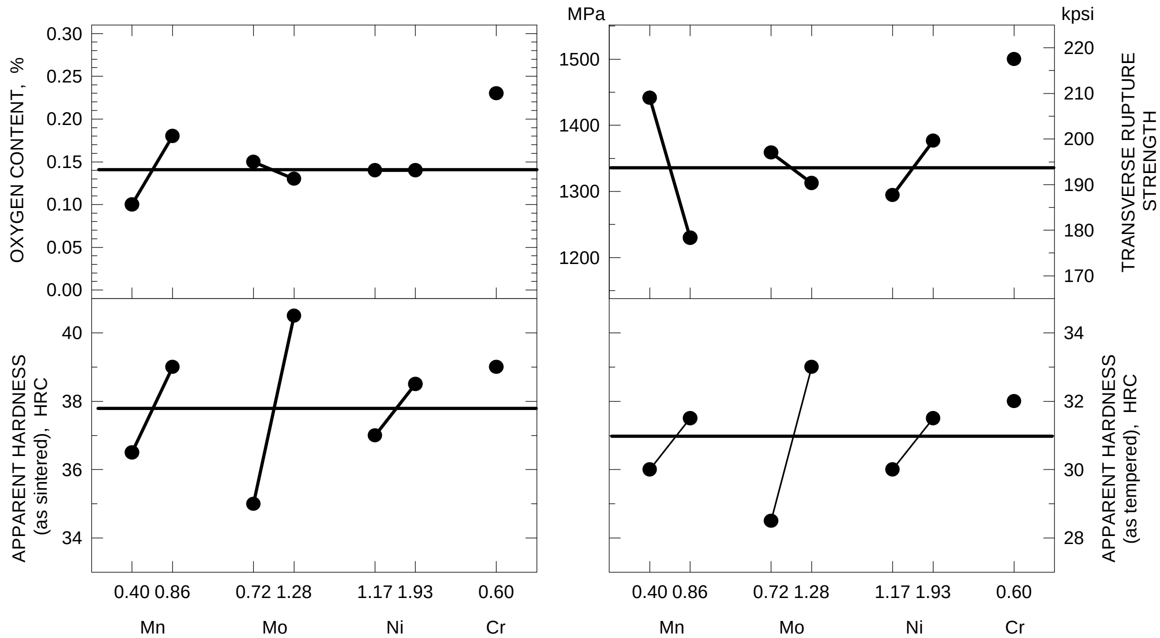

Figure 2. Mean effect of manganese, molybdenum, nickel and chromium on oxygen content of powder, transverse rupture strength, as-sintered apparent hardness and as-tempered apparent hardness.

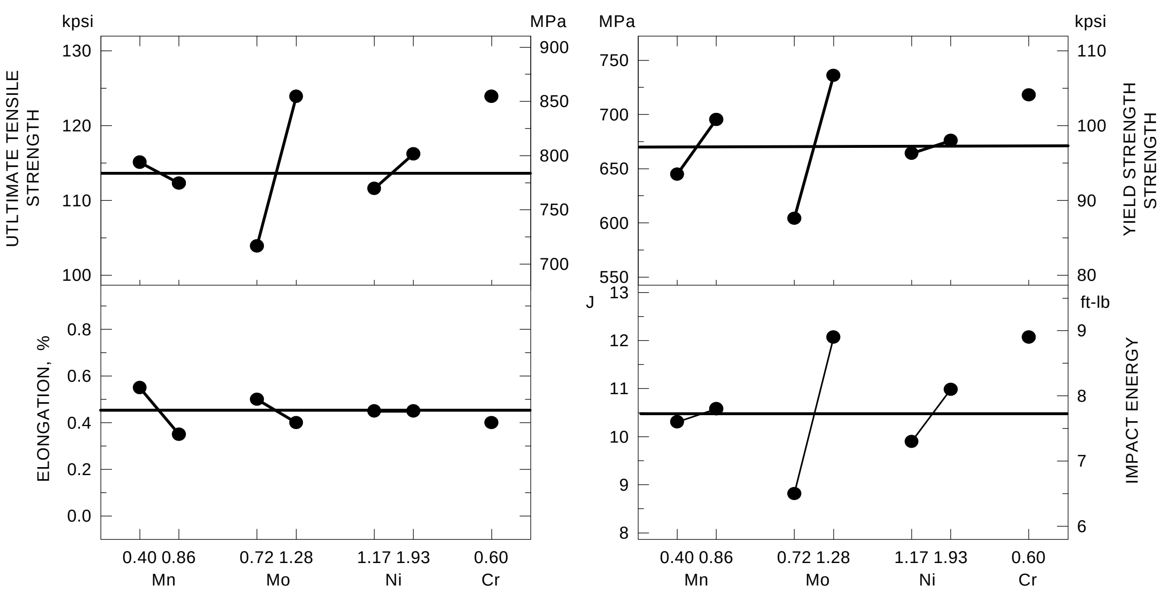

Figure 3. Mean effect of manganese, molybdenum, nickel and chromium on ultimate tensile and yield strengths, elongation and impact energy.

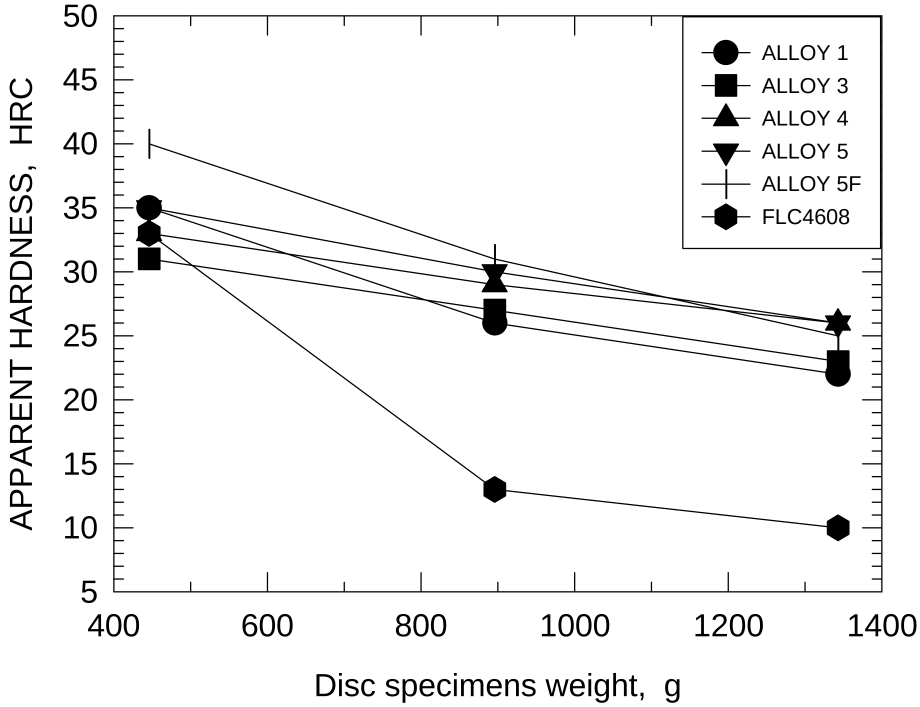

Figure 4. Effect of size on apparent hardness of the surface of disc specimens. (Average of 10 measurements on the top and bottom faces).

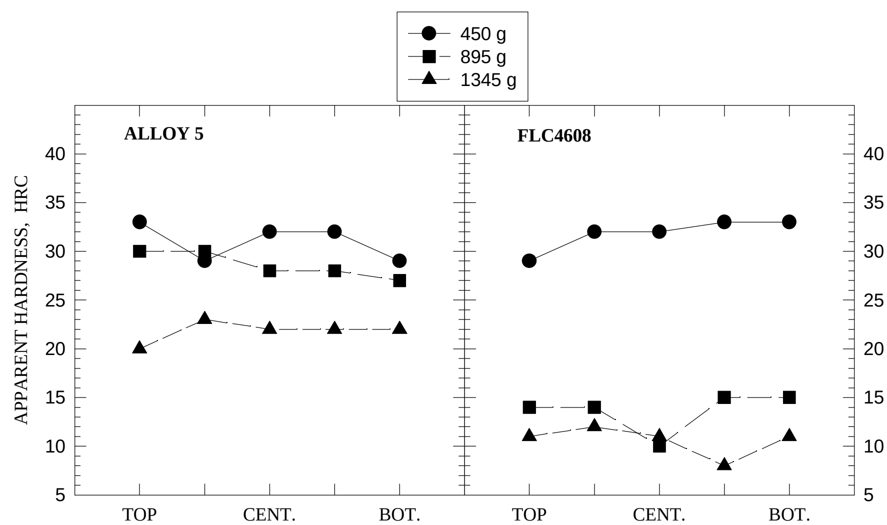

Figure 5. Apparent hardness measured on the cross section of disc specimens made from alloy #5 and a commercial FLC4608 material.

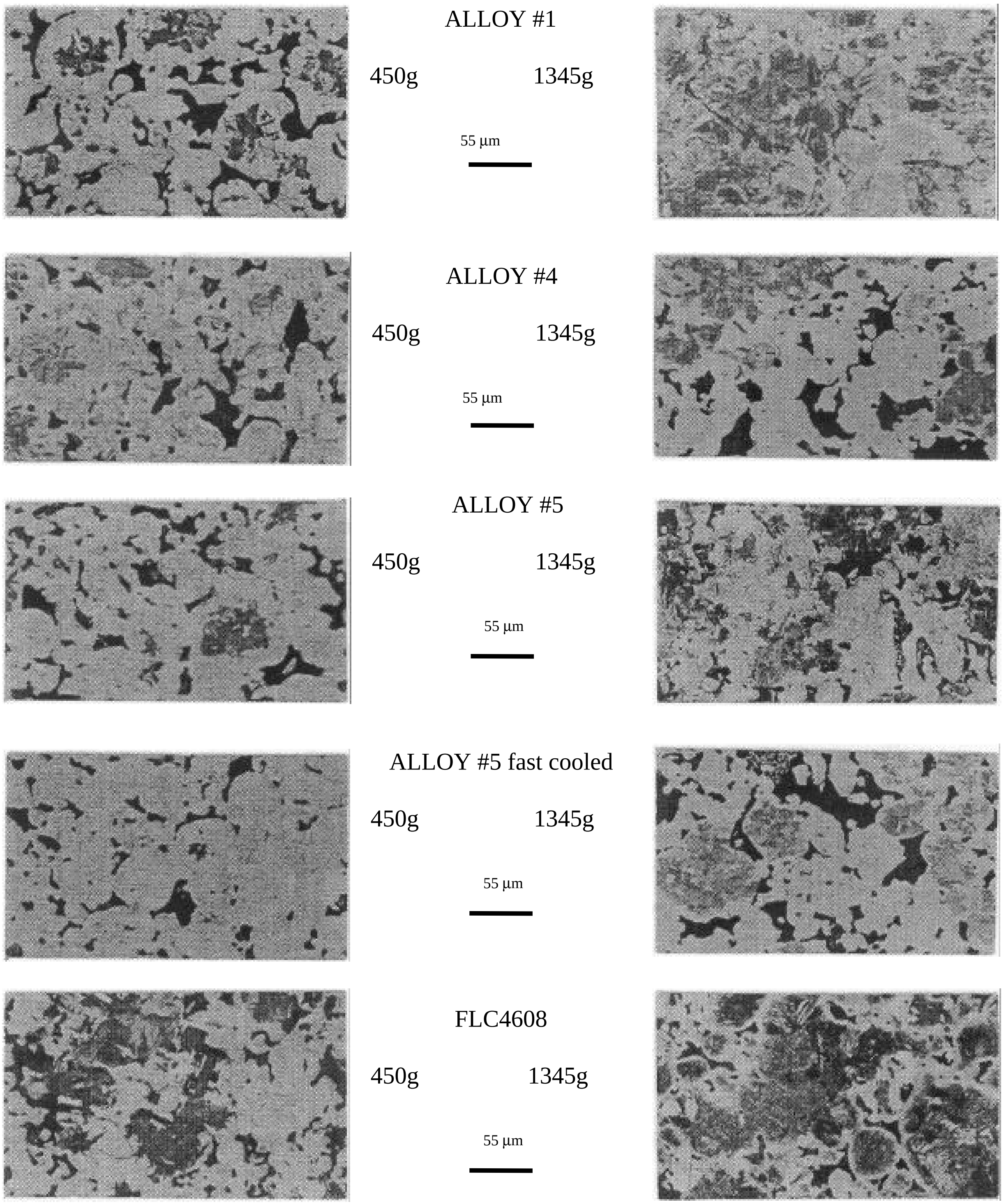

Figure 6. Microstructure of small (450g) and large (1345g) disc specimens made from alloys #1, #4, #5, fast cooled #5 and from a commercial FLC4608 mix.

oxygen content to about 0.20%. Chromium was added for its beneficial effect on hardness, strength, impact resistance and its low cost. The nickel content was fixed at 1.10% to maximize of chromium which is optimum at a Ni/Cr ratio of 2.5/1 [2]. The molybdenum was adjusted to maintain a hardenability factor of more than 25 which was achieved for a molybdenum content of 0.9%. Table 3 shows the new concentration targets for each element. The results of the validation trial are given in Table 4.

TABLE 3
Targeted chemical concentrations for the validation trial.

Mn, %	Mo, %	Ni, %	Cr,%	Hardenability factor
0.45	0.90	1.10	0.45	25.9

TABLE 4
Chemical, green and sintered properties of the validation trial.

Mn %	Mo %	Ni %	Cr %	O %	Comp. Press. tsi (MPa)	Hard. (sint.) HRC	TRS kpsi (MPa)	UTS kpsi (MPa)	YS kpsi (MPa)	Elong. %	I.E. ft.lb (J)	Hard. (temp.) HRC
0.42	0.92	1.10	0.44	0.20	35.0 (483)	36	233.4 (1610)	115.2 (794)	93.4 (644)	0.5	10.2 (13.8)	30

As expected, reducing the concentrations of manganese and chromium to 0.45% results in a significant decrease of the oxygen content to 0.2%. Also, the alloy shows a good compressibility of 35 tsi (483 MPa) at 6.8 g/cm³. This compares favorably to commercial FL4600 powders, which however have a lower hardenability factor than this alloy.

Hardness values of 36 HRC after sintering and 30 HRC were reached while transverse rupture, ultimate tensile and yield strengths of more than respectively 230 kpsi (1585 MPa), 110 kpsi (759 MPa) and 90 kpsi (621 MPa) and impact energy of 10.2 ft.lb (13.8 J) were achieved with this alloy.

CONCLUSIONS

1. A new low alloy steel powder grade was developed for sinterhardening applications. The use of manganese and chromium permitted a reduction in the amount of costly elements like molybdenum and nickel while maintaining the hardenability of the powder.

2. A combination of chromium and manganese in the range of 0.40 to 0.5 with molybdenum and nickel contents in the range of respectively 0.85 to 0.95% and 1.05 to 1.15% allowed to produce a steel powder with an oxygen content of less than 0.25% and a compressibility similar to that of FL4600 powders.

3. The high hardenability of this new alloy system allowed to readily obtained an apparent hardness of 30 HRC after conventional sintering at 1120°C and tempering.

4. The 0.45% Mn/0.45% Cr/0.90% Mo/1.10% Ni low alloy steel powder showed transverse rupture strength in excess of 200 kpsi (1379 MPa), with ultimate tensile and yield strengths of more than respectively 110 kpsi (759 MPa) and 90 kpsi (621 MPa).

5. The sinterhardening powder grade combines the high hardness and strength of heat treated materials with the high impact resistance of as-sintered materials.

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