

FACTORIAL DESIGNING APPLIED TO POWDER-METALLURGY STEELS

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Abstract

An experiment was set up to determine the critical variables in the design and manufacture of sintered steels alloyed with molybdenum, copper and nickel, whose physical and mechanical properties are important. An experimental factorial design was adopted, fractionated at two levels. The following parameters were varied: material of the matrix, percentages of carbon, copper and nickel, compacting pressure, and sintering atmosphere and temperature. The data recorded in the course of the experiment were: dimensional change, tensile strength, and sintering density, registered in ninety six test samples. The results are discussed from statistical and from metallurgical standpoints.

Keywords: powder metallurgy, statistics, factorial design, steels

INTRODUCTION

Two difficulties arise in the analysis of experiments in which many factors are involved:

1. The number of simple experiments that have to be made increases geometrically with the number of factors considered
2. Generally, only a few of the effects are found to be significant.

This means that if too many factors are taken into account, the data obtained from a large number of experiments reflect only their variability and not the essential parameters, since only a small portion of the data have any real significance.

Fractionated factorial design is a solution to this problem; it considers only a part of the whole design, this part being chosen to leave the greatest number of degrees of freedom for the determination of the low order parameters, denoting high order interactions as of zero value [1].

For a complete factorial design to determine the effect of 5 factors, $2^5 = 32$ experiments would be required, whereas a "half fraction" would need half this number – $2^{5-1} = 16$ simple experiments, whose results would be very close to those of the complete design. Half fractions (two-level fractional design) are a simple form of design but there are many other types of fractionated factorial designs, referred to generically as 2^{k-p} designs.

For example, to study the effect of 5 factors with only 16 experiments, the following procedure is adopted:

1. Write the whole factorial design for the first k-1 factors, i.e. for the factors A, B, C and D.
2. Assign the k factor to the k-1 interaction, i.e., find the column of signs for this interaction ABCD, making the column of signs E equal to that of ABCD.

In fractionated factorial designs (incomplete), several columns are repeated. Then ABC and DE interactions are said to be overlaid, or they are alias.

Like any other technique, powder metallurgy (PM) develops by scientific and experimental methods. Although it is essential, experimentation is not a guarantee of success, so obviously PM can make use of statistical methods for the design of experiments to advance, develop and attain higher standards of the quality that is expected of its products. Numerous examples in the scientific field show the benefits of factorially designed experimentation.

In any research project, properties are measured by varying the parameters that are likely to prove influential, so factorial design has an enormous number of applications since it ensures a high standard of experimentation. A number of examples are available of the use of factorial designing of products in which specific properties are required [1, 2, 3, 4] and for the improvement of procedures [2, 5] in which the maintenance of certain variables and characteristics is essential. Few studies have been published, however, on the application of factorial design to PM [6, 7].

EXPERIMENTAL

Three response variables (dependent variables) are studied in this work, chosen for their importance in the evaluation of PM steel:

1. Dimensional change.
2. Tensile strength.
3. Sintering density (measured by the Archimedes principle).

The critical factors of design and manufacture were chosen on the basis of our experience with sintered steels. The following parameters were considered important for the investigation (independent variables):

1. Composition of the matrix (Mo prealloyed content).
2. Concentration of carbon.
3. Concentration of copper.
4. Concentration of nickel.
5. Compacting pressure (uniaxial).
6. Sintering atmosphere.
7. Sintering temperature.

The upper and lower limits chosen for this design are shown in Table 1.

Tab.1. Upper and lower limits used in this factorial design.

	Factor	Lower Limit	Upper Limit
A	Matrix	Astaloy85Mo (0.85 % Mo)	AstaloyMo (1.5 % Mo)
B	% C	0.3 %	0.7 %
C	% Cu	0 %	2 %
D	% Ni	0 %	4 %
E	Pressure	500 MPa	700 MPa
F	Atmosphere	N ₂ 5H ₂	N ₂ 10H ₂ 0.1CH ₄
G	Temperature	1120°C	1180°C

The following conditions were observed in the design of the experiment:

1. A distinction was made between the populations with a minus factor and those with plus factor, and two decisions are possible for each factor of each of the response variables. The null hypothesis is that the two populations give the same measurement. The alternative hypothesis is that there is an appreciable difference between them; this

effect will have a high value and will be significant in relation to an average zero distribution.

2. The degrees of risk in selecting a not-true alternative are defined as:

- $\alpha = 0.05$, defined as the probability that a factor be taken as significant when in fact it is not,
- $\beta = 0.10$, defined as the probability that a factor be taken as insignificant when in fact it is significant.
- The criterion for choosing between the two hypotheses is based on $\Delta = 2 \sigma$.

The design chosen for the study of the above-mentioned situation is a fractionated and replicated factorial that studies 7 factors with 32 types of experiment, each with three replications to ensure adequate control of the variability of the sintered materials.

The reason for choosing a fractionated model was that a complete 2^7 factorial design needs 128 elemental experiments. If these have to be replicated three times, 384 test samples have to be prepared and tested one by one. And even with this large number of experiments, there is a disproportion between the amount of information provided and the labour involved. The 128 elemental experiments of a 2^7 design provide 7 degrees of freedom of the principal effects, and the interactions of 2 factors 21 degrees of freedom. This means that the remaining 99 degrees of freedom correspond to interactions of three or more factors which can usually be rejected. They can be used to estimate the error, but this can be done with fewer degrees of freedom, so a fractionated 2^{7-2} design is much more efficient.

The solution of this model is by $R = IV$. The model 2^{7-2} has the following generatrices:

$$F = ABCD$$

$$G = ABDE$$

For statistical analysis of the results we used the software Statgraphics.

RESULTS

The calculations of the effects produced by each of the variables on the three properties – dimensional change, tensile strength and sintering density – are shown in Table 2. In each case, the standard error is calculated with 68 degrees of freedom with the largest interactions of two factors.

The average dimensional change observed in the whole population is of 0.324 %. The effect of a factor is known to be the change expected in the response when this factor goes from a minus to a plus level. Copper is therefore seen to be one of the most significant effects since the response, i.e., the dimensional change, jumps by (0.573 ± 0.018) with the change of level, that is, when the concentration rises from 0 % to 2 % in the mix to be sintered.

The arrangement of the effects in a bar diagram, as in Fig.1, shows that the concentration of copper is by far the most significant, well ahead of that of nickel which has the opposite effect to that of copper on the dimensional change. This is no surprise since it has long been known that the addition of copper to metal alloys gives rise to swelling in the course of sintering. An addition of 4 % of nickel to powder mix produces 0.098 % shrinkage of this dimensional change in the steels of the study. Figure 1 shows also that there is less dimensional change in sintering at 1180°C than at 1120°C; according to the signs adopted, the effect is below zero. The difference expected with the change of level in this factor is of 0.070 %.

Tab.2. Effect estimation for dimensional change, tensile strength and sintering density.

Effect or Interaction	Dimensional Change [%]	Tensile Strength [MPa]	Sintering Density [g/cm ³]
Average	0.324 ± 0.009	349.6 ± 4.0	6.95 ± 0.02
A: Matrix	0.013 ± 0.018	33.8 ± 8.1	-0.02 ± 0.03
B: Carbon	-0.037 ± 0.018	47.8 ± 8.1	0.03 ± 0.03
C: Copper	0.573 ± 0.018	66.7 ± 8.1	-0.05 ± 0.03
D: Nickel	-0.098 ± 0.018	177.6 ± 8.1	0.06 ± 0.03
E: Pressure	0.049 ± 0.018	51.0 ± 8.1	0.30 ± 0.03
F: Atmosphere	0.027 ± 0.018	-0.3 ± 8.1	-0.06 ± 0.03
G: Temperature	-0.070 ± 0.018	35.5 ± 8.1	0.10 ± 0.03
AB	0.004 ± 0.018	-0.4 ± 8.1	0.02 ± 0.03
AC	0.003 ± 0.018	0.6 ± 8.1	0.05 ± 0.03
AD	-0.009 ± 0.018	8.0 ± 8.1	-0.03 ± 0.03
AE	0.006 ± 0.018	11.0 ± 8.1	0.03 ± 0.03
AF	-0.059 ± 0.018	-41.3 ± 8.1	-0.03 ± 0.03
AG	-0.024 ± 0.018	-3.3 ± 8.1	0.01 ± 0.03
BC	-0.011 ± 0.018	14.3 ± 8.1	0.06 ± 0.03
BD	0.003 ± 0.018	-22.8 ± 8.1	-0.01 ± 0.03
BE	0.001 ± 0.018	2.3 ± 8.1	-0.02 ± 0.03
BF	0.009 ± 0.018	-20.8 ± 8.1	-0.06 ± 0.03
BG	0.011 ± 0.018	14.7 ± 8.1	0.01 ± 0.03
CD	0.003 ± 0.018	-23.4 ± 8.1	0.21 ± 0.03
CE+FG	0.027 ± 0.018	-25.5 ± 8.1	-0.08 ± 0.03
CF+EG	0.046 ± 0.018	-7.3 ± 8.1	0.06 ± 0.03
CG+EF	0.020 ± 0.018	3.1 ± 8.1	0.02 ± 0.03
DE	0.063 ± 0.018	8.7 ± 8.1	0.06 ± 0.03
DF	0.005 ± 0.018	-3.0 ± 8.1	0.01 ± 0.03
DG	-0.056 ± 0.018	24.7 ± 8.1	-0.02 ± 0.03

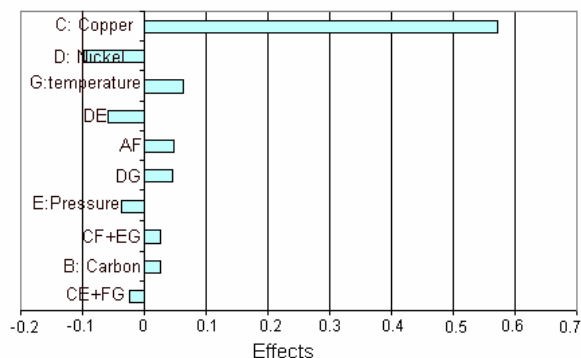


Fig.1. Arrangement, in a bar diagram, of the effects of considered factors on dimensional change.

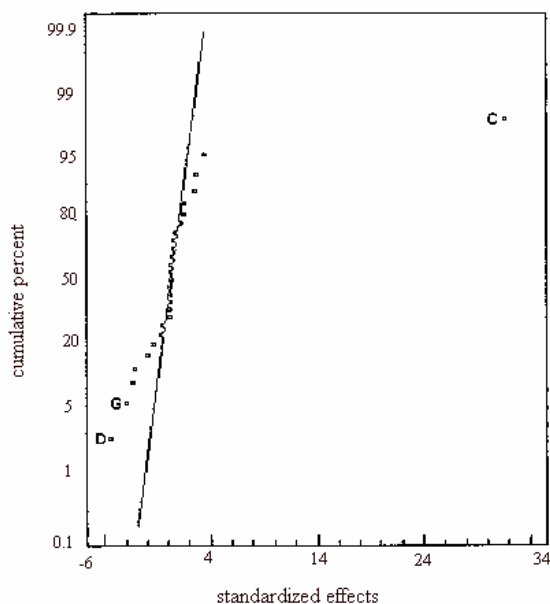


Fig.2. Representation in normal probabilistic paper of the effects of considered factors on dimensional change.

The influence of the other factors is uncertain since their estimations are more or less equal to random figures, as is shown in Fig.2. The only factors that may be taken as significant are those of C (% of copper), D (% of nickel) and G (sintering temperature) which depart from the straight line. The other estimations may be the effect of noise since they follow this line which describes a normal distribution of an average of zero and a typical deviation of 1.

The average tensile strength calculated in all the experiments is 349.6 ± 4.0 MPa. From the estimation of the effects shown in Tab.2., the nickel content is seen to be the factor that most affects the tensile strength in the steels studied. When this concentration is raised from 0 % to 4 %, the tensile strength rises by 177.598 ± 8.097 MPa.

Figure 3 is a list of all the factors in order of importance of their effect on the tensile strength. After nickel, copper is seen to be the second in importance, its response rising with its addition as an alloy, but its effect (66.7 ± 8.1 MPa) is less than half that of nickel. This is reasonable, as both of them harden the iron matrix, mainly by solid solution, and in the case of nickel the TTT curves of the steels are displaced towards the right with the consequent formation of more resistant microstructures.

The compacting pressure and the carbon content are of lesser importance, although it is obvious that a higher tensile strength is obtained with a pressure of 700 MPa than with one of 500 MPa, and with 0.7 % addition of carbon rather than with 0.3 %, since the estimations of the effects of these factors are above zero. The higher compacting pressure promotes greater density after sintering, an effect that is examined in a later paragraph, and this greater density implies lower porosity and hence a higher tensile strength. The greater carbon content promotes the formation of bainitic structures rather than pearlitic, the former being harder and stronger. In the same way, though to a lesser degree, the higher temperature in our study (which implies a greater degree of sintering) and a matrix with a high percentage of molybdenum improve the strength of the steel, since particle necks are enlarged, and hence, the Fe autodiffusion.

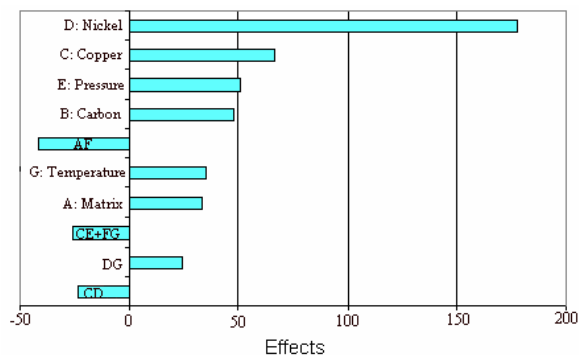


Fig.3. Arrangement, in a bar diagram, of the effects of considered factors on tensile strength.

The effect of the interaction between matrix and sintering atmosphere is worthy of mention. Its negative sign indicates that to achieve a higher tensile strength, the two factors should be opposed, i.e., that the Astaloy85Mo should be sintered with N_25H_2 or the AstaloyMo with $N_210H_20.1CH_4$. Since the factors included in the interaction are of only slight significance when considered independently, the significance lies in the interaction.

Figure 4 shows the factors that cannot be confused with those expected from random estimations. So doubt can be cast on the influence on the tensile strength of all the factors except nickel (D), copper (C), compacting pressure (E) and carbon content (B). It is curious to observe the scant influence of matrix, temperature and atmosphere of sintering, all of which can be confused with the noise attributed to a normal zero average and the typical deviation 1.

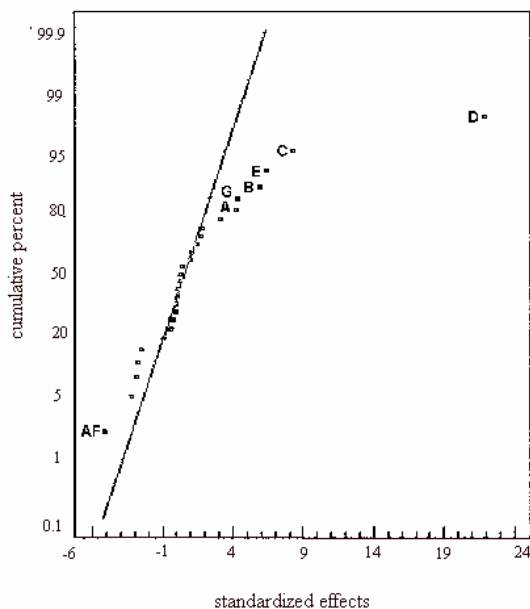


Fig.4. Representation in normal probabilistic paper of the effects of considered factors on tensile strength.

The average density after sintering in the steels under study is $6.95 \pm 0.02 \text{ g/cm}^3$. Figure 5 gives all the factors in descending order of their influence on the response, and shows how few of them have any marked influence. The most significant is the compacting pressure since it raises the density by $0.30 \pm 0.03 \text{ g/cm}^3$ when the pressure is raised from 500 MPa to 700 MPa. This is as expected as an effect of the much higher green density.

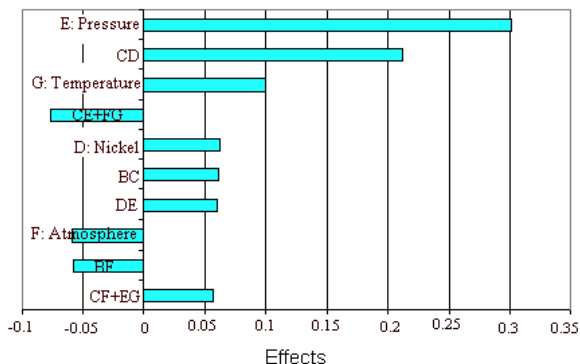


Fig.5. Arrangement, in a bar diagram, of the effects of considered factors on sintering density.

In the same Figure 5, the next most significant factor is the interaction of the percentages of copper and of nickel, which produces a variation of the sintering density up to $0.21 \pm 0.03 \text{ g/cm}^3$. It should be noted that the sign of this interaction is positive, which means that a higher density is obtained when the two factors are at the same level – that is, when neither copper nor nickel are added to the initial powder mix, or when the two are added at the same time, nickel contrasts copper effect.

The sintering temperature also affects the sintering density, although the influence is less than that of compaction pressure. The effect of temperature is $0.10 \pm 0.03 \text{ g/cm}^3$, which means that with all the other factors at a constant level, sintering at 1180°C gives a sintering density 0.1 g/cm^3 above that obtained with sintering at 1120°C . Again, this is as expected since the higher temperature promotes higher sintering and greater density.

The importance of the effect of copper on the sintering density, as compared to that of other factors, is open to doubt since it is below zero, so its addition to the powder mix produces a slightly lower density. The same can be said of the small effects of the matrix and of the carbon content.

Figure 6 shows the normal probabilistic part played by the effects of the factors, showing those that deviate in a baffling way from the straight line, such as the random variation of the noise with the normal distribution of zero average and deviation one. This analysis shows that only the factors E (compaction pressure), G (temperature) and the CD interaction (copper and nickel) can be considered influential, and this is a great help in the investigation since it means that other factors can be ruled out.

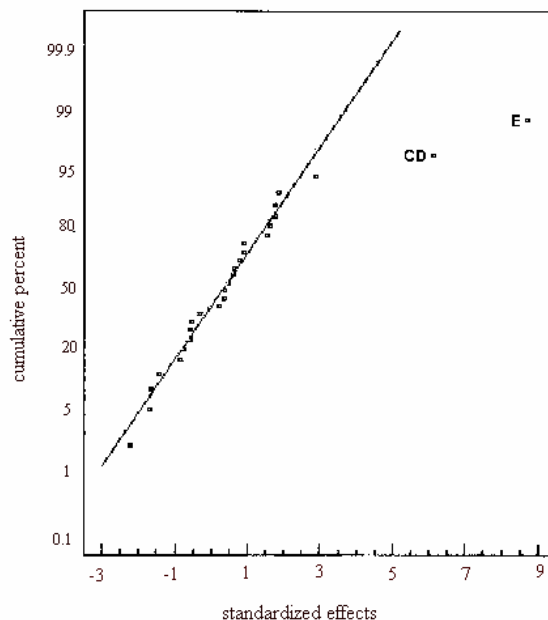


Fig.6. Representation in normal probabilistic paper of the effects of considered factors on sintering density.

In Figure 7 we see the effect of the main factor on each of the three properties. The effect of copper on dimensional change appears in Fig.7 (up). Leaving aside the importance of the other factors, one would expect a variation of 0.038 % when no copper is added, and a variation of 0.610 % with an addition of 2 %. In view of the marked influence on the dimensional change of this addition of copper, as compared to that of the other effects, any attempt to control this response could concentrate beneficially on this one factor.

As already mentioned, the percentage of nickel has the greatest effect on the tensile strength, so it is interesting to show the estimated effect when this factor passes from a minus to a plus level, or in other words, from the absence of nickel to a 4 % addition to the starting mixture, leaving the other factors constant. Figure 7 (middle) shows the change from a composition without nickel to one with a 4 % addition; the response rises from 260.8 MPa to 438.4 MPa, an increase of 68 % in the tensile strength as a consequence of the nickel content.

We have seen that the compacting pressure has an important effect on the sintering density. Leaving aside the other factors and varying only the compacting pressure, the response is given in Fig.7 (down). The density obtained when compacting at 500 MPa is 6.90 g/cm³ and rises to 7.10 g/cm³ with compaction at 700 MPa, an increase of nearly 4.5 %.

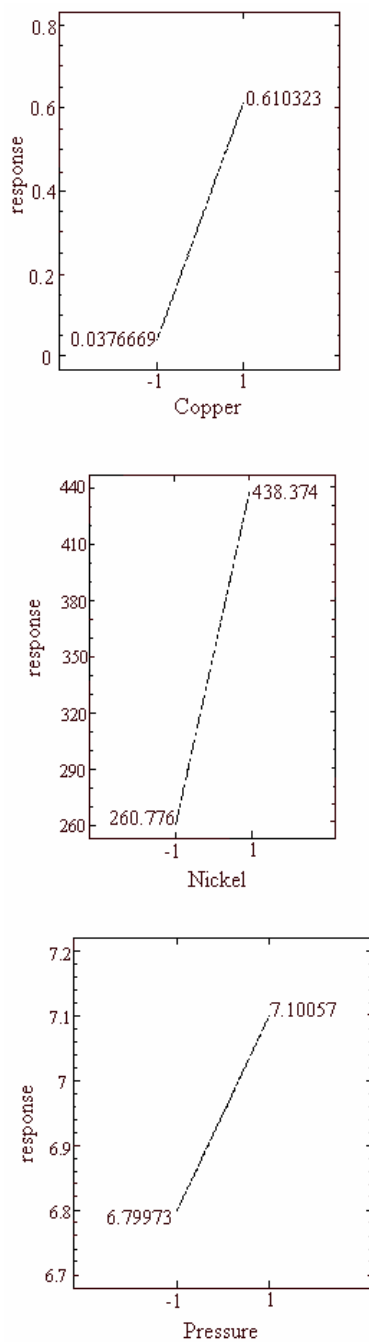


Fig.7. Effect of main factors on properties. Up) Cu percentage influence on dimensional change. Middle) Ni percentage influence on tensile strength. Down) Compacting pressure influence on sintering density.

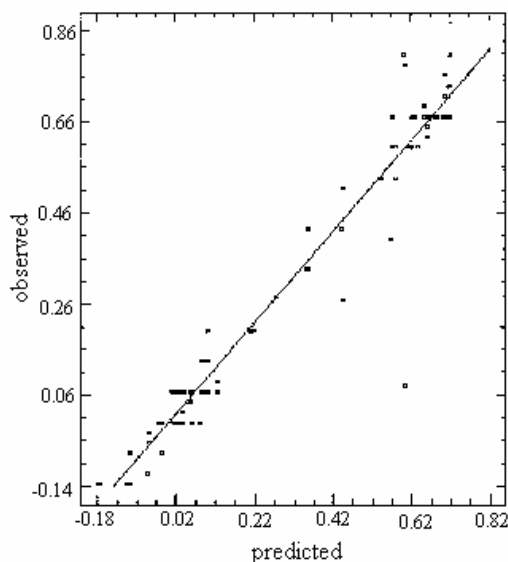


Fig.8. Graphic representation of observed vs predicted dimensional change.

The high order interactions are considered null, so sufficient data are available for a statistical analysis. The validity of the model was proved [8] by comparing the response of the observed dimensional change with that of the calculated factors (Fig.8). A good distribution of the straight line was observed of the two variables, and a graph was drawn of the residues as compared to the expected values of the response (Fig.9). No abnormality was found in the graph nor in the long-term residues (Fig.10), so the model can be taken as effective. Similar results were obtained for the other two properties.

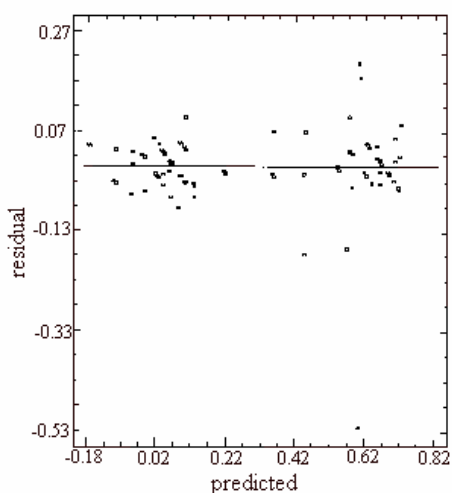


Fig.9. Graphic representation of residuals vs predicted value of dimensional change.

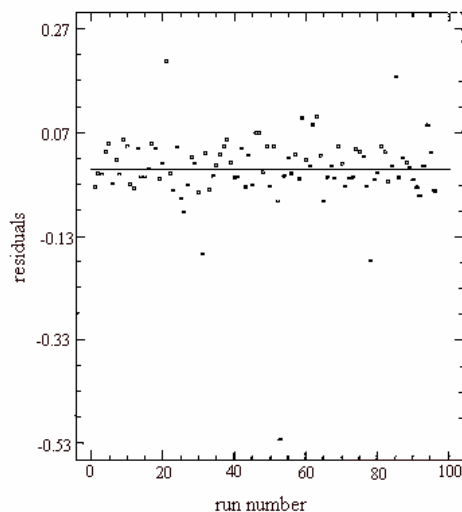


Fig.10. Graphic representation of residuals with time for predicted dimensional change.

From the 32 different materials, several were chosen for microstructural study to underline the statistical results. The effect of low carbon (0.3 %) is shown in Fig.11. The microstructures of Astaloy85Mo steel with 0.3 % carbon and without either copper or nickel, compacted at 500 MPa and sintered in $N_2 10H_2 0.1CH_4$ at 1180°C are given in Fig.11 left. The microstructure is ferrite-bainite, with a higher proportion of ferrite, due to the low (0.3 %) percentage of carbon and to the fact that some of the carbon is lost during sintering. This material has the lowest tensile strength, probably because of the higher proportion of ferrite and the low proportion of bainite. The results showed that in fact, the average resistance of the material was 188.6 MPa, while the average of all the materials of the study was 349.6 MPa.

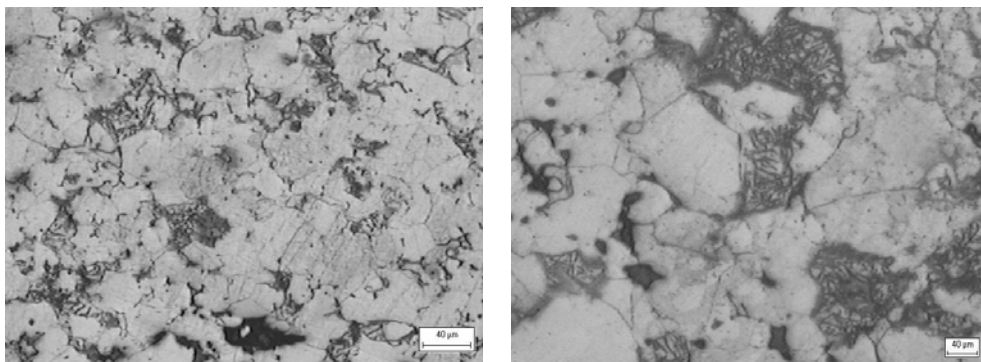


Fig.11. Left: microstructure of the steel manufactured with Astaloy85Mo matrix, 0.3 % C, no Cu, no Ni, compacted at 500 MPa and sintered in $N_2 10H_2 0.1CH_4$ at 1180°C. Right: microstructure of the steel manufactured with AstaloyMo matrix, 0.3 % C, no Cu, no Ni, compacted at 500 MPa and sintered in $N_2 5H_2$ at 1120°C.

The influence of the sintering temperature is seen in Fig.11 right - AstaloyMo steel with 0.3 % carbon and without any copper or nickel, compacted at 500 MPa and sintered in $N_2 5H_2$ at 1120°C. The bainite was higher here than in the former material.

The effect of copper on the microstructures is shown in Fig.12. AstaloyMo, with 0.3 % carbon, 2 % copper and no nickel, compacted at 500 MPa and sintered in $N_2 10H_2 0.1CH_4$ at 1120°C appears in Fig.12, left. Grains of ferrite are seen clearly, and a large amount of bainite, favoured by the high Mo content and the contribution of copper. This photograph was chosen for inclusion because of the clarity of the aureoles around the edges of the ferrite grains - a consequence of the spreading of Cu when it passes to the liquid phase. The effect described has a considerable influence on the dimensional change, which in this material was found to be 0.716 %, well above the average of 0.324 % in the other materials of the study.

Figure 12 right shows the microstructure of a matrix of AstaloyMo, with 0.7 % carbon, 2 % copper and no nickel, compacted at 500 MPa and sintered in $N_2 5H_2$ at 1180°C. The main feature of this microstructure is that, in contrast to the earlier structures shown, here there are no ferrite particles. The high percentage of Mo in conjunction with 0.7 % carbon content have swung the eutectic point enough to suppress the formation of ferrite, or to reduce its formation at the given concentration. The greater part of the microstructure is formed of bainite.

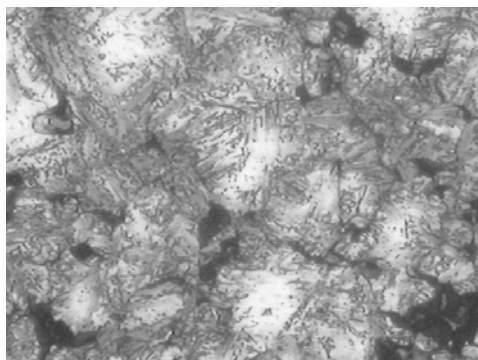
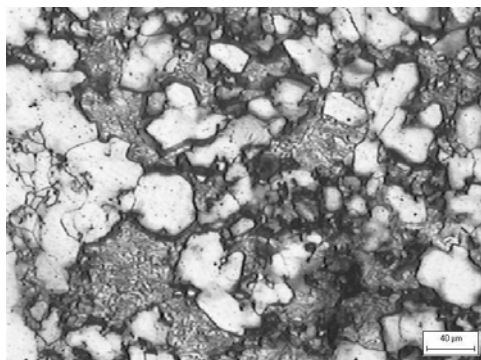


Fig.12. Left: microstructure of the steel manufactured with AstaloyMo matrix, 0.3 % C, 2 % Cu and no Ni, compacted at 500 MPa and sintered in $N_2/10H_2/0.1CH_4$ at 1120°C. Right: microstructure of the steel manufactured with Astaloy Mo matrix, 0.7 % C, 2 % Cu and no Ni, compacted at 500 MPa and sintered in $N_2/5H_2$ at 1180°C.

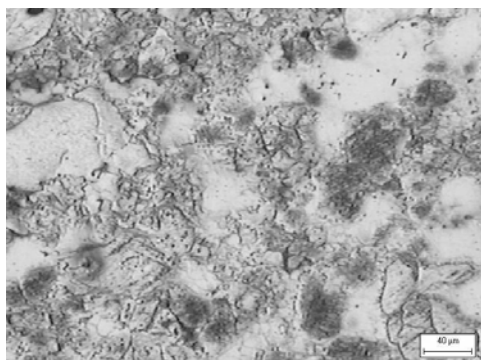


Fig.13. Microstructure of the steel manufactured with Astaloy85Mo matrix, 0.3 % C, 4 % Ni and no Cu, compacted at 700 MPa and sintered in $N_2/5H_2$ at 1180°C.

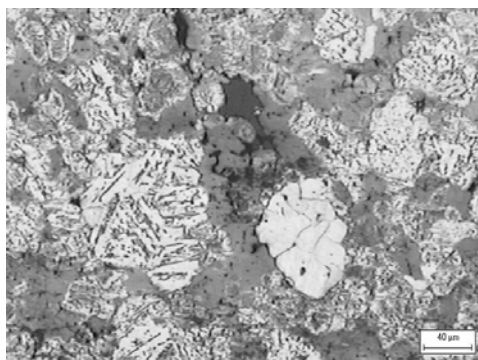


Fig.14. Microstructure of the steel manufactured with AstaloyMo matrix, 0.7 % C, 2 % Cu, 4 % Ni, compacted at 700 MPa, and sintered in $N_2/10H_2/0.1CH_4$ at 1180°C.

The effect of nickel is seen in Fig.13. As nickel is a gammagene element, it tends to stabilize the austenite phase. Large areas appear in Fig.13 with residual austenite, as well as ferrite with carbides. Finally, the microstructure in Fig.14 shows the material that has all its factors at plus level, that is, it is made from AstaloyMo powder with 0.7 % carbon, 2 % copper, 4 % nickel, compacted at 700 MPa and sintered in $N_2/10H_2/0.1 CH_4$ atmosphere at 1180°C. It contains a certain proportion of ferrite, of residual austenite and of other formations of bainite and pearlite that were difficult to distinguish with the optical microscope. This material exhibited excellent properties of strength and sintering density, both related in some way to the low porosity of the material in Fig.14, and a high percentage of bainite and martensite. The average tensile strength of this material is 480.2 MPa, well above the average of all the materials of the study which was 349.6 MPa. The average density of the material was 7.204 g/cm^3 , again above the average of the whole population, which was 6.95 g/cm^3 .

CONCLUSIONS

- Statistical experiment design in the sector of powder metallurgy provides an efficient means of determining which factors are really influential, and the complex interactions between them.
- Of all the effects studied, the concentration of copper was found to have the greatest influence on dimensional change.
- The nickel concentration was found to have the greatest influence on the tensile strength. A minor conclusion, though still important, is that the best response in regards to the tensile strength is obtained with 2 % copper, 0.7 % carbon and compacting at 700 MPa.
- Of all the effects of the study, the compaction pressure and the interaction between copper and nickel proved most influential in the sintering density.

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