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DEVELOPMENT OF OPTIMIZED DIFFUSION ALLOYED POWDERS

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ABSTRACT

Diffusion alloy powders containing molybdenum, Ni and Cu are widely used as they provide high stability, high compressibility and are very well suited for high performance applications. However, the process required to produce diffusion alloy powders (two annealing operations) makes the production of premixes with specific chemistries difficult. Moreover, recent years have seen increased volatility in alloy prices, therefore promoting the need for leaner alloys.

This paper presents the results of the study based on a FD-0205 chemistry (0.5%Mo-1.75%Ni-1.5%Cu). Design of experiments was used to optimize the powder chemistry (Mo, Ni and Cu levels) in regards to cost and to evaluate the effect of each element on static properties. These mixes were sintered in industrial furnaces with fast cooling capability in order to replicate typical sintering conditions and cooling rates.

INTRODUCTION

Diffusion-alloyed powders (DB: Diffusion bonded) containing Mo, Ni and Cu are widely used for the production of high performance parts. Their unique microstructure resulting from the partial diffusion of the nickel and copper particles during the second annealing makes them good candidates for high toughness and high fatigue applications.[1,2,3] Also, the DB process offers several advantages :

- Homogeneity : the partial bonding of Ni and Cu reduces quite significantly the segregation versus regular pre-mixes, offering enhanced part-to-part homogeneity and consistency.
- Dusting : dusting associated with Ni and Cu is almost totally eliminated[4].
- Compressibility : nickel and copper being only partially diffused during manufacturing, the compressibility remains excellent, which allows production of high density parts.

However, with the high volatility of alloy prices encountered in the past decade, the need for the production of leaner alloys has gained a significant importance. Indeed, alloys such as Ni and Mo have more than doubled in price in recent years[5]. This has resulted in a search for lower cost alternatives to the

production of high alloyed high performance parts such as micro-alloying or use of non-traditional alloys (Nb, V,Ti, Si and Mn)[6,7,8], production of higher density materials[9,10] or production of leaner alloys[11].

It is clear that the existing DB grades are not so competitive costwise and development of leaner versions of grades with similar properties is required. However, unlike standard pre-mixes, where chemistry can be adjusted as required by the customer at the blending stage in order to reach the required properties, the number of different versions of DB powder is extremely limited as a consequence of process and logistic limitations. Another avenue is the use of DB master powders, which can be diluted to achieve the required properties at the lowest possible cost. This avenue was already investigated and results under conventional cooling conditions were published in a previous paper[12] in 2012.

The objective of this work was to develop lower cost alternatives to standard diffusion alloyed powders like ATOMET DB46, a diffusion alloyed powder containing 0.50% pre-alloyed Mo and 1.50% Cu and 1.75% Ni diffusion bonded (FD-0205). The effect of Mo, Ni and graphite on the green and sintered properties was established and used to build a model able to optimize parts properties with regards to cost. In order to limit the amount of diffusion bonded powders required, a master diffusion alloyed powder richer in Ni and Cu than the mixes required for the study was produced. This master alloy was used to produce mixes containing different amounts of Ni, Mo and Cu by dilution with base powders containing various levels of Mo. TRS and tensile properties were evaluated under fast cooling conditions.

EXPERIMENTAL PROCEDURE

A master diffusion alloyed mix was produced in the laboratory based on a chemistry similar to a FD-0205 but enriched three times with Ni and Cu. This mix containing about 4.0% Cu and 5.3% Ni diffusion bonded as well as 0.5% pre-alloyed Mo was used as the base powder for the study. This powder was then mixed with different amounts of ATOMET 1001, ATOMET 4001, ATOMET 4401 and ATOMET 4901, steel powders containing 0, 0.55%, 0.85% and 1.50% pre-alloyed Mo, respectively. The chemistry of the base powders used is in Table 1.

Table 1 - Chemistry of the base powders

Powder	Cu (%)	Ni (%)	Mn (%)	Mo (%)
DB master	4.0	5.3	0.14	0.50
ATOMET 1001	-	-	0.20	-
ATOMET 4001	-	-	0.15	0.55
ATOMET 4401	-	-	0.15	0.85
ATOMET 4901	-	-	0.15	1.50

Design of experiments (DOE) was used in order to reduce the number of analysis needed. L9 Taguchi arrays were built using Mo, Ni and graphite content as the three parameters. The Mo concentration was lowered by the addition of ATOMET 1001 (1001) while it was increased by the addition of either ATOMET 4401 (4401) or ATOMET 4901 (4901). ATOMET 4001 (4001) was used for the dilution of Ni and Cu at the Mo content of 0.5%. Ni content was governed by the amount of DB master powder used in the mix. Graphite was admixed to the desired level to complete the Taguchi array. Cu content also varies in the different mixes but was not included in the arrays as it always varies linearly with Ni and is therefore not an invariable parameter. Cu levels were 0.3%, 0.75% and 1.1% corresponding to Ni levels of 0.4% 1.0% and 1.5%. The effect of Cu will be taken into account during analysis of the Taguchi arrays.

Four L9 Taguchi arrays were analysed in this study. The three levels used for each parameter are shown in Table 2. The Taguchi arrays are labeled T1 to T4. The number in parenthesis corresponds to the steel powder grade used to dilute the DB master mix to reach these levels of Mo.

Table 2 – Levels used for the four Taguchi arrays

T1		
Mo (%)	Ni (%)	Graphite (%)
0.35 (1001)	0.4	0.4
0.55 (4001)	1.0	0.5
0.75 (4401)	1.5	0.6

T2		
Mo (%)	Ni (%)	Graphite (%)
0.35 (1001)	0.4	0.4
0.55 (4001)	1.0	0.5
1.25 (4901)	1.5	0.6

T3		
Mo (%)	Ni (%)	Graphite (%)
0.35 (1001)	0.4	0.4
0.55 (4001)	1.0	0.6
0.75 (4401)	1.5	0.8

T4		
Mo (%)	Ni (%)	Graphite (%)
0.35 (1001)	0.4	0.4
0.55 (4001)	1.0	0.6
1.25 (4901)	1.5	0.8

A total of 16 mixes were required to build these four Taguchi arrays, as some of the mixes were used for more than one array. A cost equation was determined using alloying composition, processing and additives costs. Then, a cost index was calculated for all mixes compared to a standard ATOMET DB46. The average alloy prices of Q4 2012 were taken for the study. A cost of 1 represents a mix with a cost identical to ATOMET DB46. Cost indexes below 1 show mixes less expensive than ATOMET DB46 and above 1 mixes more expensive than ATOMET DB46. The chemistry of the mixes is listed in Table 3. All mixes were produced with KS15 graphite and admixed with 0.75% zinc stearate (ZnSt).

Table 3 – Chemistry of the mixes used in this study

Mix identification	Mo	Ni	Graphite	Cost index
A	0.35	1.5	0.4	0.80
B	0.35	1.0	0.6	0.72
C	0.35	0.4	0.5	0.63
D	0.55	1.5	0.5	0.83
E	0.55	1.0	0.4	0.75
F	0.55	0.4	0.6	0.66
G	0.75	1.5	0.6	0.87
H	0.75	1.0	0.5	0.78
I	0.75	0.4	0.4	0.69
J	1.25	1.5	0.6	0.94
K	1.25	1.0	0.5	0.86
L	1.25	0.4	0.4	0.76
M	0.35	0.4	0.8	0.63
N	0.55	1.5	0.8	0.84
O	0.75	1.0	0.8	0.79
P	1.25	1.0	0.8	0.86

Rectangular bars of 31.7 mm X 12.7 mm X 6.35 mm (1.25 in X 0.5 in X 0.25 in) and tensile samples (dog bones[13]) 89.64 mm long by 6.35 mm thick (3.529 in X 6.35 in) were pressed to 7.0 g/cm³. The samples were sintered in an industrial furnace with fast cooling capability. The samples were sintered at 1125°C for 20 minutes and cooled at a rate of 2.1°C/s in the range of 650°C to 315°C. Half of the samples was evaluated in the as-sintered condition while the other half was subjected to a tempering treatment at 205°C for one hour in air.

Properties such as compressibility, green strength, dimensional change, transverse rupture strength (TRS), tensile properties and apparent hardness were evaluated. The mechanical properties were evaluated both in the as-sintered and tempered states, as described earlier.

Results obtained from the analysis of these data were used to evaluate the effect of the different alloying elements on mechanical properties as well as possible interactions between the three alloying elements. These Taguchi arrays were also used to build a model, which is able to optimize the mechanical properties of a mix vs. its cost, finding cost effective mixes with properties similar to those of a reference mix of ATOMET DB46. These mixes were then produced and tested under the same conditions to compare predicted values to the actual experimental values.

Results

L9 Taguchi arrays at 0.4, 0.5 and 0.6% graphite (T1 and T2)

Figure 1 shows the effect of Mo, Ni and graphite on the compressibility and on sintered properties such as dimensional change, TRS, apparent hardness and UTS of the master DB diluted with ATOMET 1001, ATOMET 4001 and ATOMET 4401 with 0.4, 0.5 and 0.6% graphite (Taguchi T1 plan). In all graphs, the red dotted line represents the average value of all nine mixes included in the Taguchi array. The green dotted line is the value for an ATOMET DB46 reference sintered under the same conditions. In this case the reference is taken as ATOMET DB46 admixed with 0.6% graphite.

Very little impact of the three alloying elements is observed on compressibility. The overall range is only 15 MPa (~ 1 TSI). In regards to dimensional change, Ni appears to have the biggest impact. According to Figure 1, an increase in Ni results in an increase in dimensional change. In this mix, it would be expected that Ni would induce shrinkage, meaning that as Ni is increased the dimensional change would decrease. However, as mentioned earlier, an increase in Ni content also results in an increase in Cu content (the two vary linearly with the amount of DB master mix). This increase in dimensional change observed on Figure 1 could be explained by the increase in Cu occurring with the increase in Ni. The mechanical property graphs (TRS, UTS and apparent hardness) show similar tendencies. Indeed, as expected, an increase in all alloying elements results in an increase in mechanical properties. The impact of increasing the Mo content from 0.35% to 0.75% was very similar to that of increasing the Ni content from 0.4 to 1.5% (and correspondingly, Cu from 0.3 to 1.1%).

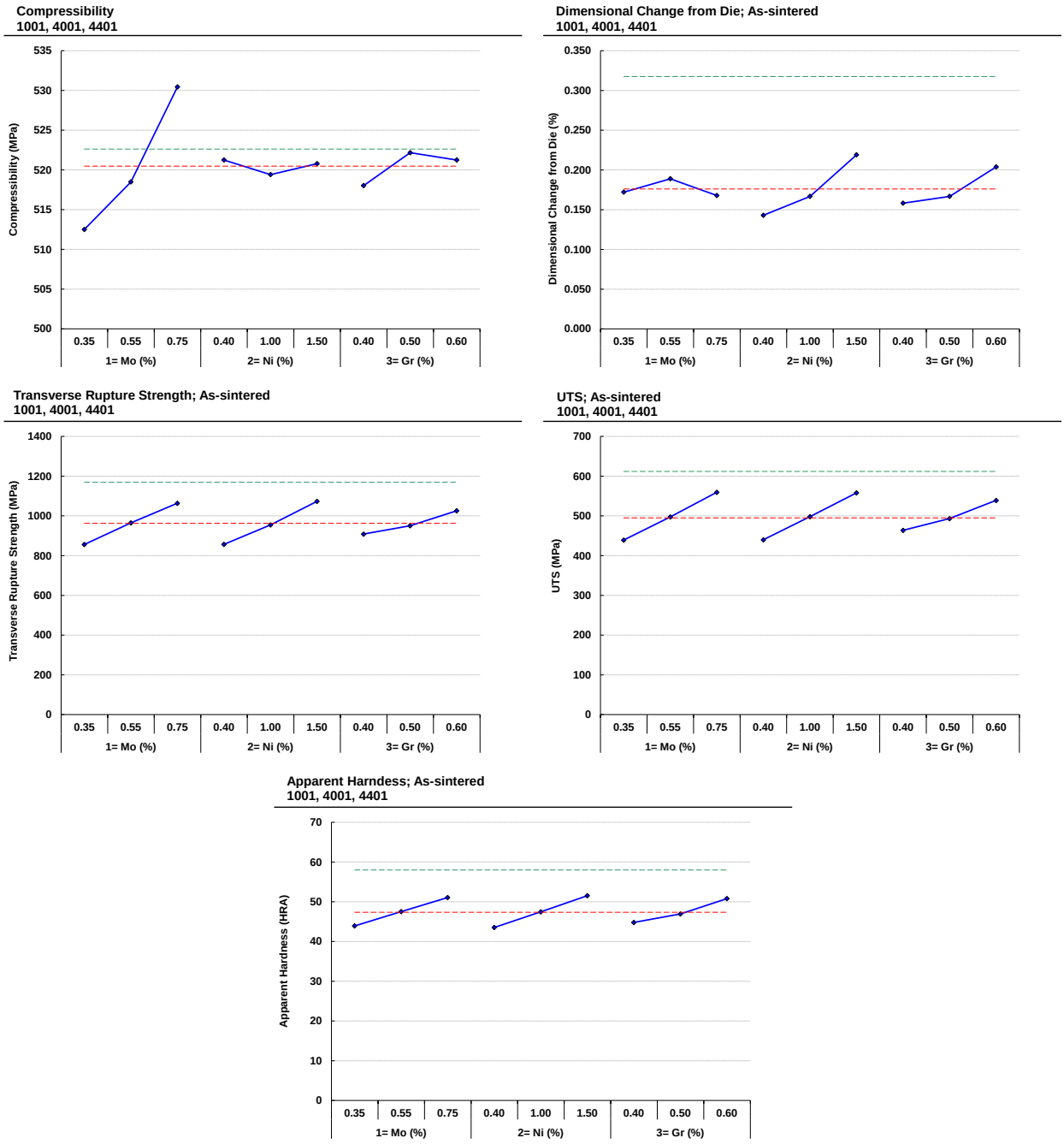

Figure 1 - Effect of Mo, Ni and graphite on green and sintered properties of mixes containing ATOMET 1001, 4001 and 4401 (Taguchi T1)

Figure 2 shows the same analysis but replacing mixes of ATOMET 4401 with ATOMET 4901. This means that the three levels of Mo are 0.35, 0.55 and 1.25% (instead of 0.75%). Similar trends as with the previous results are obtained except that as the Mo range increases, so does its impact. Indeed, regardless of price, it appears that increasing Mo to its highest level will have the most impact on mechanical properties.

In both cases, increasing the level of Ni from 0.4% to 1.5% resulted in a difference of about 225 MPa on the TRS. A similar impact is also observed with the graphite ranges of the two Taguchi arrays. This means that the two Taguchis are complementary. Since L9 arrays can only contain 3 levels for each parameter, they were treated separately. However, if the impact of Mo is plotted combining the four levels versus TRS as depicted in Figure 3, a linear trend is observed with the first three levels of Mo and then flattens out before reaching 1.25%. This means that the level of 1.25% may be overdesigned. Using a powder with 1.0 or 1.1% could be sufficient.

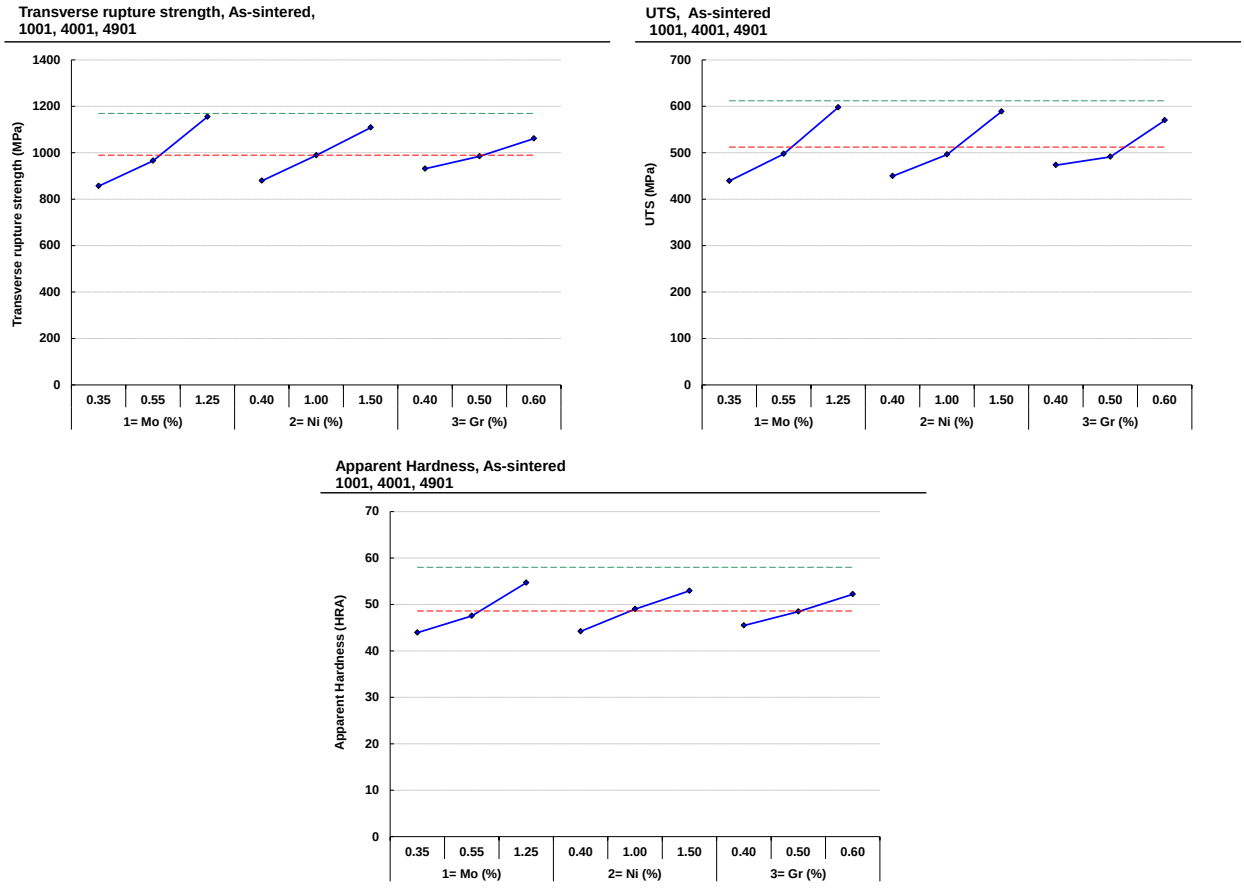

Figure 2 - Effect of Mo, Ni and graphite on sintered properties of mixes containing ATOMET 1001, 4001 and 4901 (Taguchi T2)

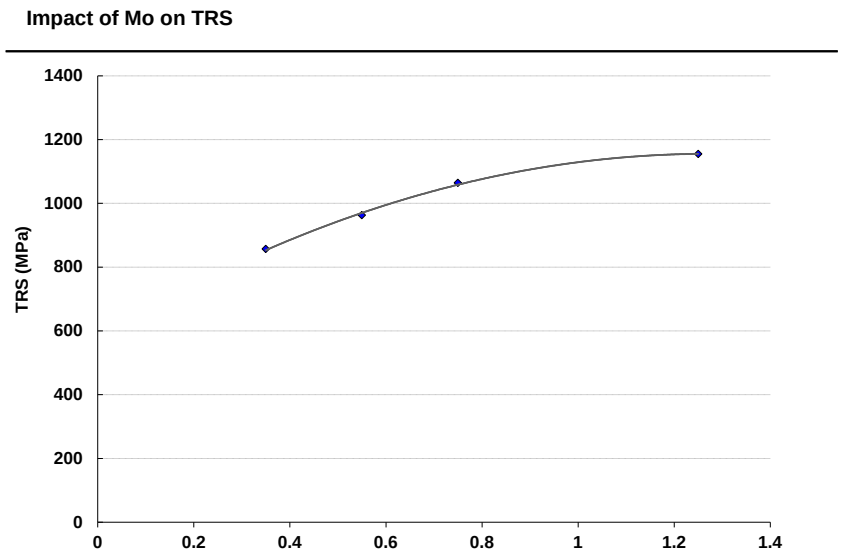

Figure 3 – Impact of Mo on TRS (T1 and T2 combined)

L9 Taguchi arrays at 0.4, 0.6 and 0.8% graphite (T3 and T4)

Figure 4 shows the results of the Taguchi array performed at levels of 0.4, 0.6 and 0.8% graphite with Mo levels of 0.35, 0.55 and 0.75% (ATOMET 1001, ATOMET 4001 and ATOMET 4401) (T3). In regards to compressibility, very little difference is observed with the previous series of data except that graphite has a larger impact given its wider range.

On the other hand, significant differences are observed on the mechanical properties. As expected, an increase in Mo and Ni still results in an increase in mechanical properties as observed on the TRS and UTS graphs of Figure 4. However, as graphite increases, a downward shift appears on the TRS and the increase of the UTS is reduced. This is explained by the greater amount of untempered martensite formed at higher levels of graphite in these fast cooling furnaces.

L9 Taguchi arrays allow the evaluation of three or four parameters. When only three parameters are used, it is possible to evaluate the interactions between the other parameters. The interaction estimated for mechanical properties such as TRS and UTS could not be ignored in the sintered state. These interactions are illustrated for example by the fact that an increase in Mo from 0.35 to 0.55% results in very similar strength. This means that in certain mixes an increase in the Ni content combined with an increase in Mo results in lower strength. In the as-sintered state this could be explained by the greater amount of untempered martensite formed at high levels of alloying and high carbon content. In this case, the interaction of parameters accounted for 23% on TRS, almost the same as all three parameters and 15% on the UTS, making it impossible to predict a mechanical property based on these parameters. This phenomenon was not observed in the previous study[12] as the differences in microstructure, and their effects were not as important when sintered with slower cooling conditions.

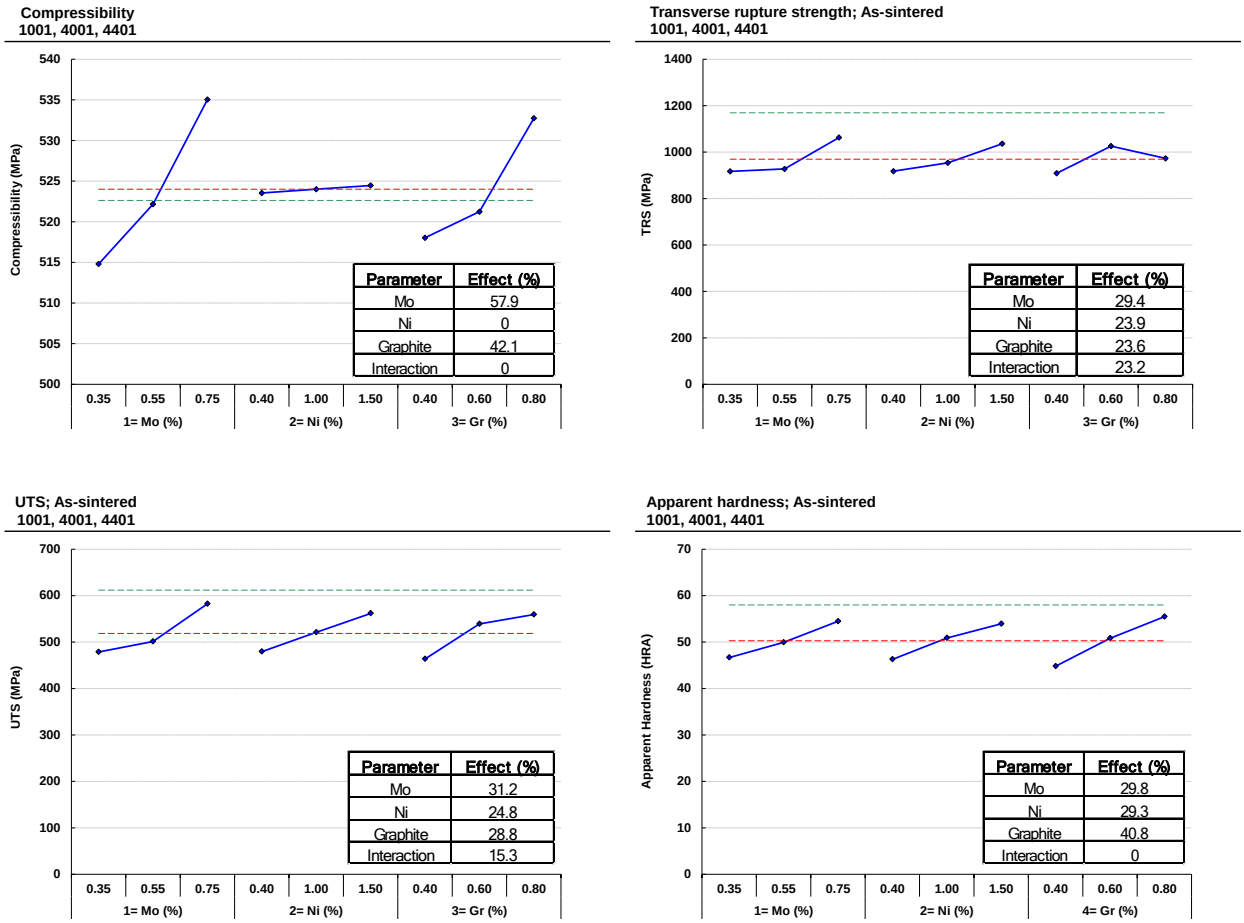

Figure 4 – Effect of Mo, Ni and graphite on sintered properties of mixes containing ATOMET 1001, 4001 and 4401. The effect of all parameters and their interactions are indicated in the tables. (Taguchi T3)

In this case, tempering will be required to improve TRS and UTS at 0.8% graphite and reduce the interactions encountered between alloying elements. Figure 5 represents the results obtained with the same mixes after tempering. It can be noted that in all cases no interaction is present in these conditions. An increase in Mo, Ni and graphite results in increases in mechanical properties.

A similar trend is obtained when using mixes at 1.25% Mo (T4) instead of 0.75% (graphs not included). In the as-sintered mixes, the interaction between parameters is also very strong and requires tempering to be able to predict the effect of each parameter on mechanical properties.

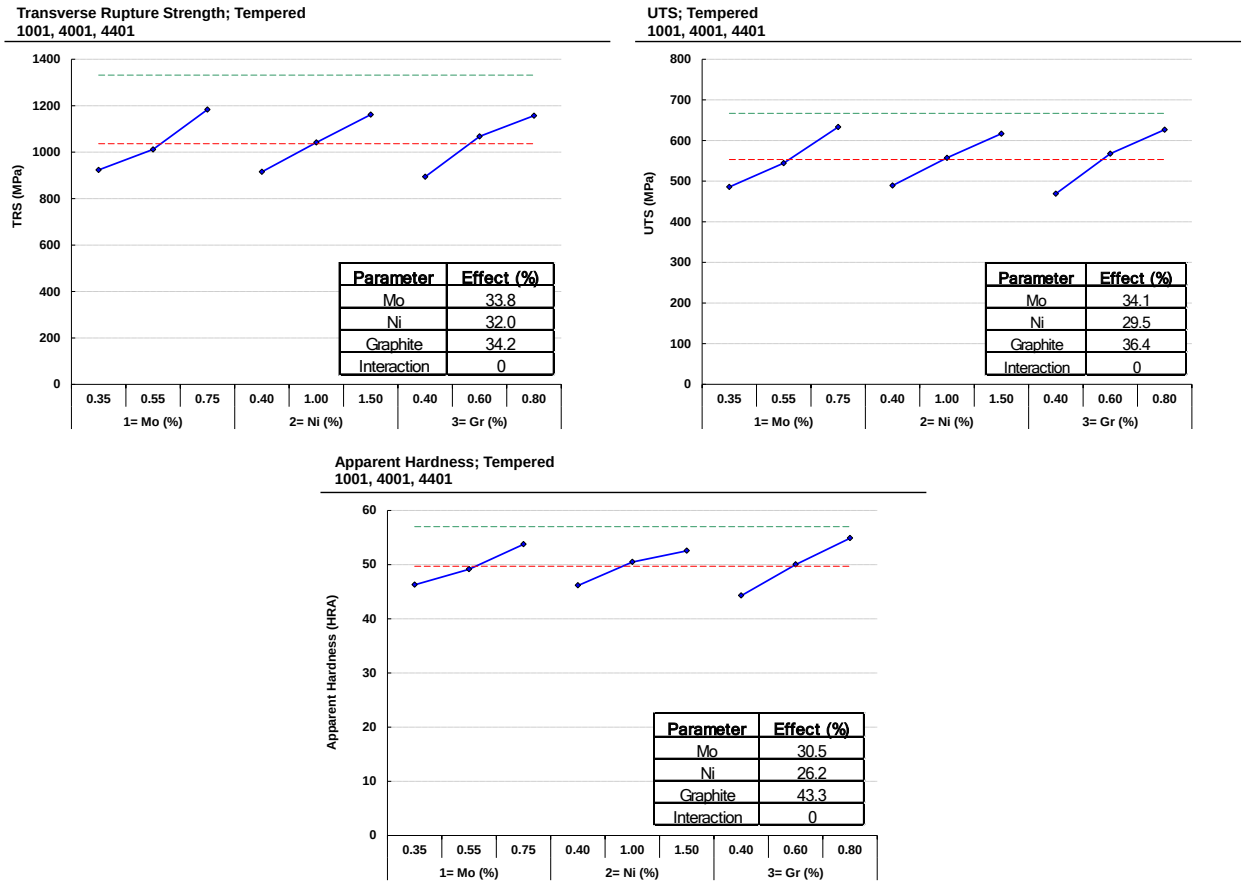

Figure 5 – Effect of Mo, Ni and graphite on tempered properties of mixes containing ATOMET 1001, 4001 and 4401 (Taguchi T3)

Although all microstructures are very typical of DB structures, they varied greatly with the composition. Figure 6 shows as-sintered microstructures for a low alloy mix (Mix C: 0.35% Mo, 0.4% Ni, 0.5% gr.) and a more highly alloyed mix (Mix J: 1.25% Mo, 1.5% Ni, 0.6% gr.) compared to a ATOMET DB46 reference at 0.6% graphite. As depicted in Figure 6A, mix C (which is the mix containing the lowest amounts of Mo, Ni and only 0.5% graphite) has a mixture of pearlite and ferrite in its microstructure. Mix J which is the mix containing the highest amounts of Mo, Ni and 0.6% graphite is made of a network of Ni rich areas with a matrix of untempered martensite and small areas of pearlite. The Ni-rich areas are expected to contain some martensite and retained austenite. When raising the cooling rate, more martensite was formed in the high alloyed materials than in a previous study performed at low cooling rates[12].

The reference material is constituted of a matrix of pearlite with Ni rich areas.

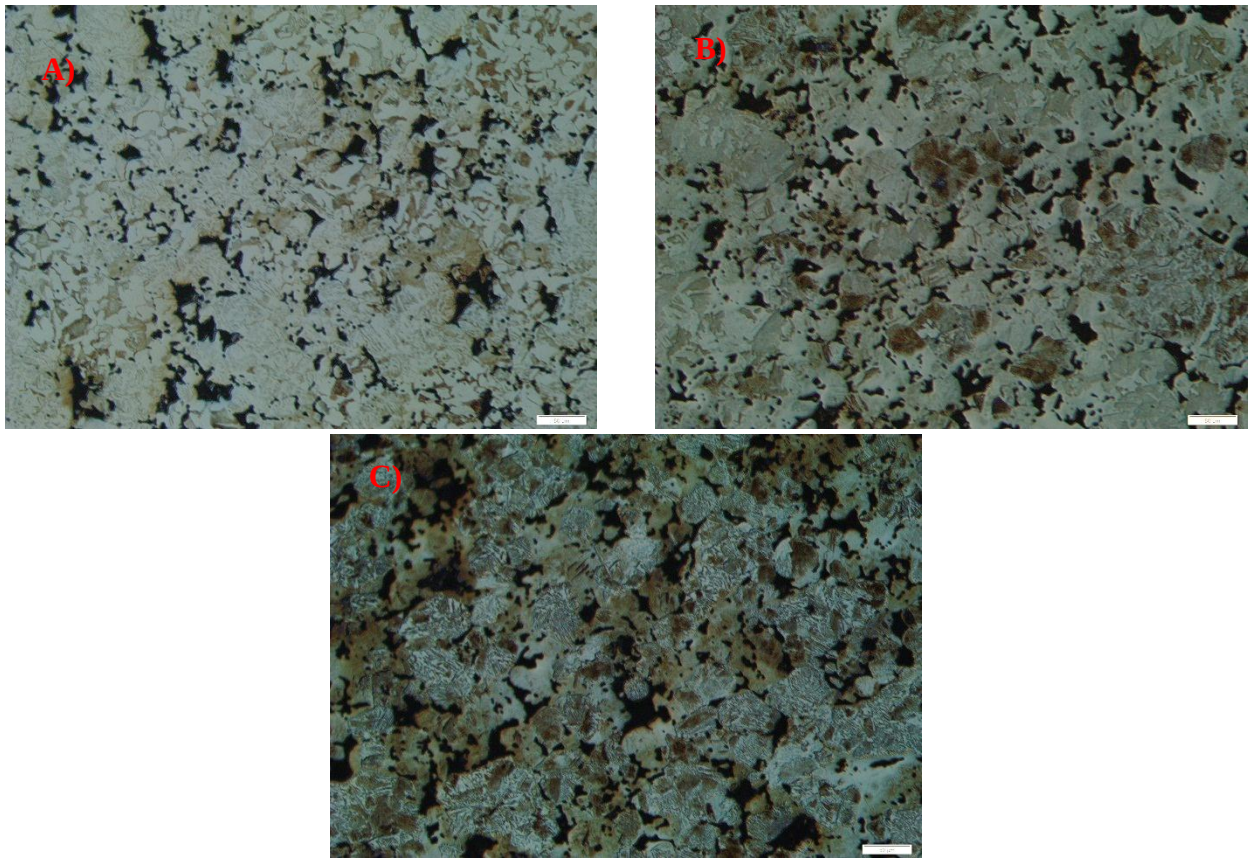

Figure 6 – As-Sintered Microstructures of A) Mix C (0.35% Mo, 0.4% Ni, 0.5% gr.) B) Mix J (1.25% Mo, 1.5% Ni, 0.6% gr.) C) ATOMET DB46-REF (0.6% gr.)

Optimum mixes prediction

Based on the results obtained with the four Taguchi arrays, equations were drawn to predict each property vs. the three parameters used (Mo, Ni, gr). A model was built with these equations for TRS, apparent hardness, UTS and dimensional change. The cost equation elaborated to determine the cost ratio of each mix was set as the target for the optimization of these properties. The objective was to obtain mixes with similar mechanical properties as the ATOMET DB46 references with 0.4, 0.6 or 0.8% graphite in the as-sintered or tempered states. The references were produced with the same additives as the test mixes; 0.4, 0.6 and 0.8% graphite KS15 and 0.75% ZnSt. The properties of the reference mixes are listed in Table 4.

Table 4 – Properties of ATOMET DB46 reference mixes

Mix	As-sintered				Tempered			
	Dim.Ch. vs. Die (%)	TRS (MPa)	Apparent Hardness (HRA)	UTS (MPa)	Dim.Ch. vs. Die (%)	TRS (MPa)	Apparent Hardness (HRA)	UTS (MPa)
DB46 0.4%	0.3	1136	51	562	0.3	1155	51	573
DB46 0.6%	0.3	1181	58	609	0.3	1314	57	660
DB46 0.8%	0.3	883	63	509	0.3	1365	60	679

In order to match the as-sintered properties, the Taguchi arrays with 0.4%, 0.5% and 0.6% graphite (T1 and T2) were used. In order to match the properties of the mixes with 0.8% graphite, tempering was required and therefore the Taguchi arrays with levels of 0.8% graphite (T3 and T4) were used. Limits of ± 25 MPa, -2.5/+5 HRA and -35/+75 MPa were set in the program for TRS, hardness and UTS respectively. Consequently, mixes found were similar to the references or better. No limit was set for the dimensional change as most mixes were significantly more negative than the reference mixes. New tools would have to be designed in order to use such mixes. Mix chemistries matching the as-sintered properties of the references are in Table 5. Since the four base powders cannot be used in the same analysis, two mixes per reference were determined; one per Taguchi array. Mixes 1 and 3 were determined using T1 while mixes 2 and 4 using T2. In all cases, it is possible to see that the Mo and carbon levels are always at the maximum permitted by the model. This suggests that Mo is the most cost effective element in regards to mechanical properties. Even though Mo alone is more expensive, its positive impact on properties appears greater than Ni and Cu.

Table 5 – Mix chemistries matching properties of the reference mixes in the as-sintered conditions

Reference matched	Array	Mix	Mo (%)	Ni (Cu) (%)	Graphite (%)	Cost Index
DB46 – 0.4% gr.	T1	Mix 1	0.75	0.88 (0.66)	0.6	0.77
	T2	Mix 2	1.25	0.42 (0.32)	0.6	0.77
DB46 – 0.6% gr.	T1	Mix 3	0.75	1.12 (0.85)	0.6	0.81
	T2	Mix 4	1.25	0.65 (0.49)	0.6	0.80

In the case of the tempered solutions, the Taguchi arrays T3 and T4 were used. As was the case in the as-sintered properties, Mo content is maximized in all cases. Mixes 5 and 6 show the efficiency of the three elements (Mo, Ni and graphite) in increasing mechanical properties while minimizing costs. The program first maximizes Mo. It then increases graphite until it reaches the required mechanical properties. When graphite is also maximized, Ni and Cu are increased (mixes 7 to 10). This means that Mo and graphite are the most effective solutions to strengthen the materials at lower cost. However, this may be because only three different levels of Mo were available. Since the model is able to find solutions with similar prices by increasing either Mo or Ni and Cu, it is possible that the optimum solution could have been found with Mo content between that of 0.75 and 1.25%

Table 6 – Mixes chemistry matching properties of the reference mixes in the tempered conditions

Reference matched	Array	Mix	Mo (%)	Ni (Cu) (%)	Graphite (%)	Cost Index
DB46 – 0.4% gr. T	T3	Mix 5	0.75	0.40 (0.30)	0.73	0.69
	T4	Mix 6	1.25	0.40 (0.30)	0.59	0.76
DB46 – 0.6% gr. T	T3	Mix 7	0.75	0.92 (0.69)	0.80	0.77
	T4	Mix 8	1.25	0.48 (0.36)	0.80	0.78
DB46 – 0.8% gr. T	T3	Mix 9	0.75	1.17 (0.88)	0.80	0.82
	T4	Mix 10	1.25	0.73 (0.55)	0.80	0.82

In all cases, mixes resulting in cost savings of about 20% or more were identified with similar predicted properties. The mixes suggested by the model were produced and processed using the same conditions as previous mixes. The values measured on the samples were compared to those predicted by the model to ensure good correlation and confirm the effectiveness of the model. The results for as-sintered and tempered TRS are in Figure 7 and Figure 8. The mix numbers on the graphs refer to those of Table 5 and Table 6. All mixes had measured values similar to the predicted values. The largest difference is seen on mix 8 and it is of only 3%. Similar results were obtained with UTS and hardness. This confirms the precision of the model.

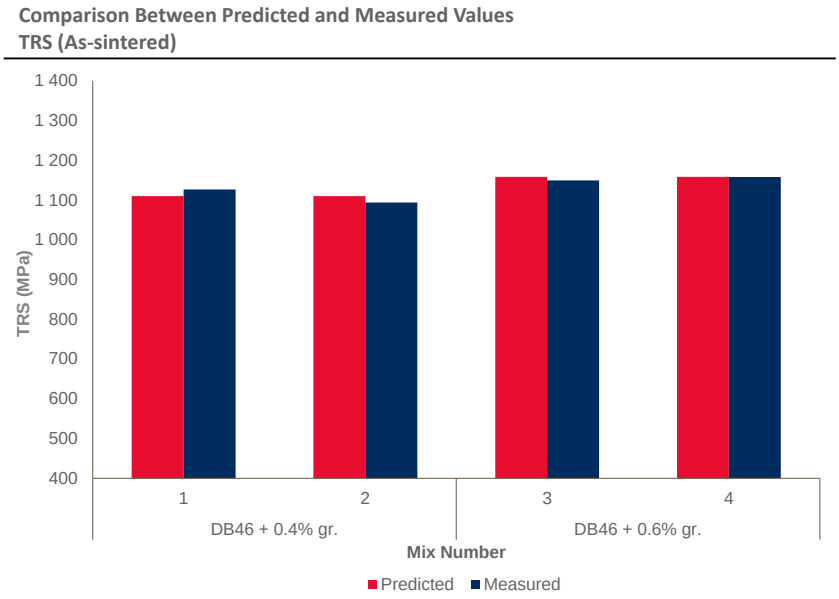

Figure 7 – Comparison between TRS values predicted and measured for as-sintered mixes.

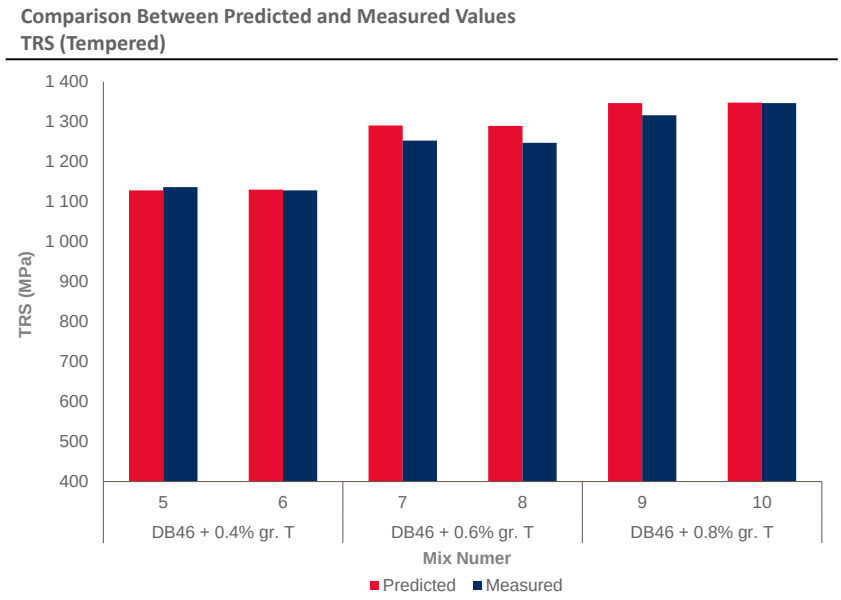

Figure 8 – Comparison between TRS values predicted and measured for tempered mixes.

Conclusion

A study was carried out in order to develop diffusion bonded powders with the same or better mechanical properties at lower cost than the actual powders available on the market. This was achieved by producing a master alloy used to elaborate mixes with different levels of Mo, Ni, Cu and graphite.

- A model was built to predict properties of diffusion bonded mixes with regards to Mo, Ni and graphite content.
- Mixes with cost ratios of 0.77 to 0.81 with properties similar to as-sintered ATOMET DB46 with 0.4 and 0.6% graphite were identified.
- Mixes with cost ratios of 0.69 to 0.82 with properties similar to tempered ATOMET DB46 with 0.4, 0.6 and 0.8% graphite were identified.
- A maximization of the Mo is the most cost effective solution in the levels tested (with current prices of alloying elements) to obtain leaner alloys with high mechanical properties followed by an increase in graphite.
- The impact of Mo on TRS seams to flatten out above 0.8% Mo, therefore the use of 1.25% Mo may be overdesigned for the objectives of this project. Further study is required to optimize the Mo content between that of ATOMET 4401 and ATOMET 4901.
- The level of Ni and Cu were fixed by the ratio present in the ATOMET DB46. The avenue of using two different master alloys, one with Cu and one with Ni could be investigated to dissociate the impact of the two elements on mechanical properties.

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