High effectiveness of the organic-bonding process in reducing the Ni, Cu and Fe dusting in a production environment

Thomas, Yannig; Pelletier, Sylvain; St-Laurent, Sylvain; Gélinas, Claude

HIGH EFFECTIVENESS OF THE ORGANIC-BONDING PROCESS IN REDUCING THE NI, CU AND FE DUSTING IN A PRODUCTION ENVIRONMENT

Yannig Thomas, Sylvain Pelletier, Sylvain St-Laurent* and Claude Gélinas*

Industrial Materials Institute, National Research Council Canada,
Boucherville, Québec

* RTIT Powder Division
Tracy, Québec

ABSTRACT

The binder-treatment blending technique is very effective in bonding fine particles of graphite, metallic additives and/or lubricants to the coarser iron particles. Main advantages compared to conventional mixes are better flow, improved productivity, part consistency and reduced segregation. The dusting is also significantly reduced compared to conventionally blended mixes. Recent developments in the FLOMET™ process, which is a proprietary binder-treatment technique, significantly improved the bonding efficiency of fine Ni and Cu particles. The bonding strength of these additives is now of the order of that achieved with the diffusion-bonding process.

In this study, the bonding efficiency of binder-treated 1.5Mo-4Ni-2Cu powders was evaluated on an industrial press and compared to that of regular unbonded and diffusion-bonded powders, through the measurement of their dusting resistance and the air quality of the workplace atmosphere. The sampling method used for measuring the amount of dust (Ni, Cu, Fe and others) on the press operator and inside the press close to the feeding zone is described. Results are compared to airborne exposure limits especially regarding the nickel dust. The compacting and ejection behavior of these various powders are also presented.

INTRODUCTION

Powder metallurgy is a highly developed method of manufacturing reliable ferrous and non ferrous parts from elemental powders such as iron, mixed with additional alloying powders such as graphite, nickel and copper. During handling and pressing, fine additives are more prone to segregate and could potentially generate hazardous dust. Since several years, more and more emphasis has been placed in the P/M industry to control and minimize exposure to powders in the atmosphere. The P/M community is at the present time very aware of that with the new European Union regulation REACH (Registration, Evaluation, Authorisation and Restriction of Chemicals) that requires that all the powder and part producers pre-register between June and December 2008 and assess the risk for the environment and human health of all substances manufactured and/or imported in Europe.

In order to reduce segregation and the potentially hazardous dust, in particular in the case of mixes containing a lot of alloying elements, as well as to improve pressing productivity and/or part-to-part consistency, the diffusion-bonding as well as the organic binder treatment processes were introduced many years ago. The first process consists in annealing the iron admixed with Cu and Ni particles in order to partially diffuse these elements to the surface of iron particles. The second process consists in using organic products to chemically bond alloying additives, graphite, lubricant and others to the surface of iron powders during the blending. One obvious advantage of the organic-bonding technique is the fact that no diffusion of Ni and Cu is involved, resulting in no change in compressibility of the base powder.

Recent developments in the binder-treatment technique significantly improved the bonding efficiency of fine Ni and Cu that achieves now the same order as the one achieved with the diffusion-bonding process [1]. This paper proposes to assess the risks by inhalation for workers using such mixes as compared to regular ones using a typical exposure scenario by compacting long runs of parts on an industrial compaction press. More precisely, the amount of dust (Ni, Cu, Fe and inhalable dust) generated by different types of powder mixes of the ferrous system 1.5%Mo-4%Ni- 2%Cu and collected on the press operator as well as close to the pressing zone was monitored and compared. The compacting and ejection behavior of these various powders is also presented.

OCCUPATIONAL EXPOSURE LIMITS AND SAMPLING CRITERIA FOR AIRBORNE PARTICULATE MATTER

Occupational Exposure Limits

Occupational Exposure Limit values (OELs) are set by competent national authorities or other relevant national institutions as limits for concentrations of hazardous compounds in workplace air, ideally using the concept of "no observed adverse effect levels". They are often expressed as a time-weighted average value (TWA) and are defined as “the concentration of a substance to which most workers can be exposed without adverse effect averaged over a normal 8-h workday or a 40-h workweek.” The OELs are usually defined under the assumption that a worker can be exposed to a substance for a working life of 40 years with 200 working days per year. The limits may arise from cases of human exposure, experiments, or epidemiological studies of exposure-response relationships. Others come from the results of animal studies.

The OELs emitted by the Occupational Safety & Health Administration in USA for different substances present in P/M mixes are given in Table 1. They vary from 1 mg/m^3 for nickel and copper to 10 mg/m^3 for iron. On the other hand, OEL values could vary as a function of the countries or national institutions that emitted these limits. As shown in Table 2 for metal nickel species, the OEL is at 1 or 1.5 mg/m^3 in USA while it differs from one country to the other in the European Union (EU). The reasons for such differences in OELs include divergence in the assessment methods and different assessments on the actual risks of the chemicals themselves. For metal nickel, it will certainly take some time before the EU Scientific Committee proposes a final OEL. In the meantime, the OEL value for Ni of 0.5mg/m^3 can be considered as a conservative value to assess the risks for EU workers. It is worth mentioning that in regards to registration to REACH, and as part of the health risk assessment, a more precautionary exposure limit called Derived No Effect Levels (DNELs) should be given instead of OELs [2], except when the routes, duration and frequency of exposure are similar to that covered by the OEL method.

Table 1. Occupational Exposure Limits for Ni, Cu, Fe (OSHA, USA)

OEL / PEL Permissible Exposure Limit (OSHA, USA)	TWA-8h, mg/m^3
Nickel	1
Copper	1
Iron	10

Table 2. Occupational Exposure Limits (TWA, 8h) for nickel metal for different countries and institutions

Countries	Institution or Ministry that emit the OELs	Name	mg/m^3	Note
USA	OSHA	PEL (Permissible Exposure Limit)	1	Total dust
USA	ACGIH	TLV (Threshold Limit Value)	1.5	Inhalable fraction
Germany		MAKs (Maximale Arbeitsplatz Konzentrationen)	0.5	Inhalable fraction
France	INRS	VME (Valeurs limites de Moyenne d'Exposition)	1	All forms
Spain	INSHT	VLA-ED (Valor Límite Ambiental Exposición Diaria)	1	All forms
Sweden	AFS	NGV (nivågränsvärde)	0.5	Total fraction
United Kingdom	HSE	MEL (maximal exposure limit)	0.5	All forms
China	MH	PC-TWA (Permissible Concentration)	1	
Taiwan	TCLA	TWA	1	
Korean	ML	TLV	1	

OSHA (Occupational Safety & Health Administration); ACGIH (American Conference of Governmental Industrial Hygienists); HSE (Health & Safety Executive); INSHT (Instituto Nacional de Seguridad e Higiene en el Trabajo, or National Institute for Occupational Safety and Health); INRS (Institut National de Recherche et de Sécurité, or National Institute of Safety Research); TCLA (Taiwan Council of Labor Affairs); ML (Ministry of Labor); MH (Ministry of Health), GBZ-2 document.

Sampling criteria for airborne particulates

Regarding to powder metallurgy activities, dust inhalation is usually the most important human exposure route. Dust can be split into three size ranges which have different effects on the respiratory system of a human being. The smaller the particle the deeper into the lungs it penetrates, where it may settle out onto the lining. Historically, respirable dust has required most of the attention, but in the last few years, the effect of larger sizes of particles on the upper respiratory tract, throat, nose and mouth have been investigated. Thoracic dust affects the respiratory tract from the thorax down and contains therefore respirable particles. Inhalable dust affects the complete respiratory tract and therefore contains both thoracic and respirable particles. Figure 1 shows particle size distribution of typical alloying additives in ferrous elemental powder mixes and their potential hazardous effect in the respiratory tract if inhaled.

For nickel species, the American Conference of Governmental Industrial Hygienists (ACGIH) as well as several institutions in European Union such as the Health & Safety Executive (HSE) in the United Kingdom have determined that the OELs should be based on the “Inhalable particulate” fraction of airborne workplace exposures. Several evaluation methods for metallic dusts in the workplace atmosphere exist such as NIOSH 7300/ OSHA ID-125G [3,4], and MDHS 42/2 [5] (specifically for nickel and inorganic compounds of nickel in air). They describe the collection and subsequent analysis of metal particulates by spectroscopic analytical techniques such as ICP-AES (inductively coupled argon plasma - atomic emission spectroscopy).

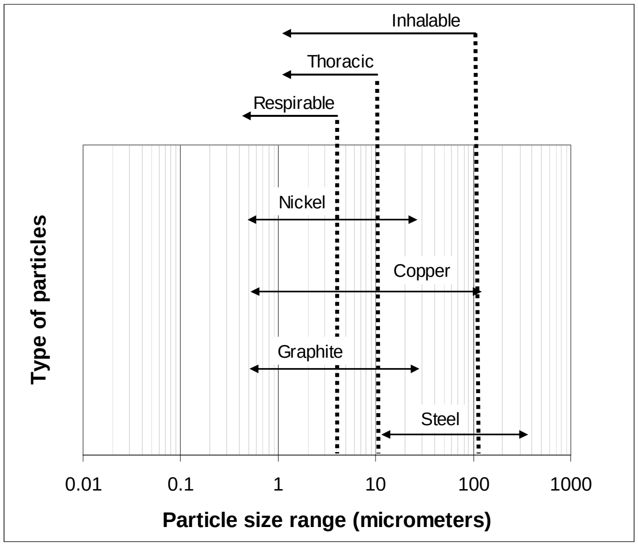

Figure 1. Potential hazardous effect in the respiratory tract if inhaled of typical PM ferrous alloying additives

EXPERIMENTAL PROCEDURES

I. Materials Description and Characterization

Four different powder mixes based on the same ferrous alloy system 1.5Mo-4Ni-2Cu (FLDN4C2-4900, according to MPIF material designation) were prepared in a Patterson Kelley twin shell V-Type blender (working volume ~28 liters (1 cubic feet)). This blender is equipped with devices for injection and drying of organic binding solution, allowing production of binder-treated mixes in accordance with the FLOMET™ technology [6,7]. Figure 2 shows the processing and blending routes for the four materials used in this study:

- ⇒ CM: consists in dry mixing in a conventional manner the Mo steel alloy, ATOMET 4901 from QMP (RTIT Powder division), with copper, nickel (Inco, grade T123PM), 0.6%wt graphite and 0.8%wt ACRAWAX C.
- ⇒ DBM: consists in dry mixing in a conventional manner similar amount of graphite and ACRAWAX C with the diffusion-alloyed powder ATOMET DB49, where copper and nickel were partially diffused to ATOMET 4901 during an annealing treatment.
- ⇒ OBM and OBM-F: consist in dry mixing in a conventional manner 0.6%wt of graphite and 0.7%wt ACRAWAX C to binder-treated powders containing ATOMET 4901, copper and fine nickel produced in accordance with the FLOMET organic-bonded technology developed by QMP. OBM and OBM-F contain respectively a fine nickel, grade T123PM and an extra-fine nickel, grade T110D. Both nickel powders are produced by Inco Limited and have a D50 particle size of 8µm and 1.5µm respectively.

In all cases, the graphite and lubricant were added and admixed with the same method in order to evaluate the benefit of the diffusion and organic bonding treatments on the bonding efficiency of copper and nickel particulates vs. a conventional un-bonded mix.

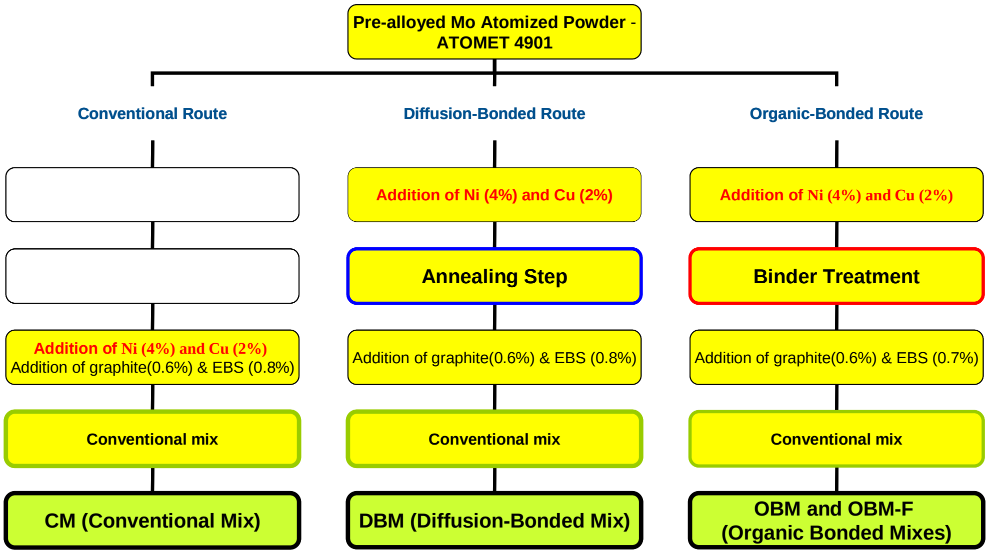

Figure 2. Description of the four materials used in this study

The particle bonding efficiency was evaluated through 2 methods: A- Evaluation of distribution of nickel and copper powders by X-ray chemical mapping with a Hitachi scanning electron microscope (SEM); B- determination of the dusting resistances of the carbon, copper and nickel using an elutriation method, described in details in [8]. For method B, 25 or 50 g of powder mix was placed in a 2.5 cm diameter steel tube and fluidized with a strong air flow of 6.0 liters/minute for five minutes. This causes the dust to be entrained as a result of a large surface-to-volume ratio and, in the case of graphite, low specific gravity. The mixture remaining on the screen plate was then analyzed to determine the relative amount of carbon, copper and nickel remaining in the powder after dust test. The dusting resistance for carbon, copper and nickel is expressed as a percentage of the initial mix concentrations. It is worth mentioning that the carbon dusting resistance corresponds not only to the graphite but also the organic lubricant. The apparent densities and flow rates of the four powder mixes were also determined according to MPIF Standards 03 and 04.

II. Air Quality Measurement of the Workplace Atmosphere

The exposure scenario used represents typical parts manufacturing in a P/M production facility. For this study, long runs of about one thousand of ¾ inch height sprockets having a density of 7.0g/cm^3 were produced using the four different powder mixes: the conventional mix CM, the diffusion-bonded mix DMB and the two organic bonded mixes OBM and OBM-F. An example of sprockets produced in this study is shown in Figure 3.

Figure 3. Sprockets produced in this study (2" O.D., 0.75" Height, 7.0 g/cm^3)

The objective of air monitoring is usually to determine the worker exposure to dust, and therefore the various sampling methods (NIOSH, OSHA, HSE,…) were developed specifically for personal sampling in the breathing zone. In this study, the objective was also to evaluate the bonding efficiency of the various pre-mixes routes by measuring the dust generated very close to the pressing area. Therefore, the following sampling strategy was used by setting samplers:

- *On the press operator*: the samplers were fixed to the lapel of the worker, in the breathing zone and as close to the mouth and nose as practicable, and the sampling pump was attached around the waist (Figure 4a)
- *In fixed locations close to the compaction area inside the press*. However, to take into account possible fluctuations of air displacement in a production environment, it was decided to place samplers at three different locations as shown in Figure 4b. Results reported in the next sections are the average measured with these three samplers.

Calibrated personal sampling pumps, SKC AirLite model 110100 (Houston, Tx), and preloaded inhalable IOM filter cassettes with 0.8µm cellulose ester membrane [9] were used to collect in the workplace atmosphere inhalable dust up to 100µm in equivalent aerodynamic diameter, following typical procedures described in [3,4,5].

Sampling was done during the production of ~800-1000 sprockets on an industrial 150 ton Gasbarre mechanical press. Parts were compacted at a stroke rate of 10 parts/min. With a sampling rate close to 2 liters per min (recommended for dust sampling), the air volume that was collected through the pumps was close to the 200-250 liters. This is half of the value recommended by the OSHA method for the measurement of the time weighted average (TWA). However, these compaction conditions were chosen following preliminary compaction tests to prevent over loading of the filters in the feeding zone of the press (below 2 mg of total dust). For reproducibility evaluation, the air sampling was done twice on two long compaction runs for the regular mix CM and the organic bonding mix OBM.

After sampling, a protective cover was set, and the cassette/filter assemblies were sent for analysis to a certified laboratory (Galson Laboratories, Syracuse, NY). The amount of inhalable dust was evaluated by the weight gain of the filters (modified NIOSH method 0500). Further analyses of the filters for the Fe, Cu and Ni particulates were carried out using modified NIOSH 7300 /OSHA 125G methods [3,4]. Two unused filters from the same lot of filters used for sample collection were also submitted for analysis. The same handling procedure was used with these blank filters, but no air was drawed through them.

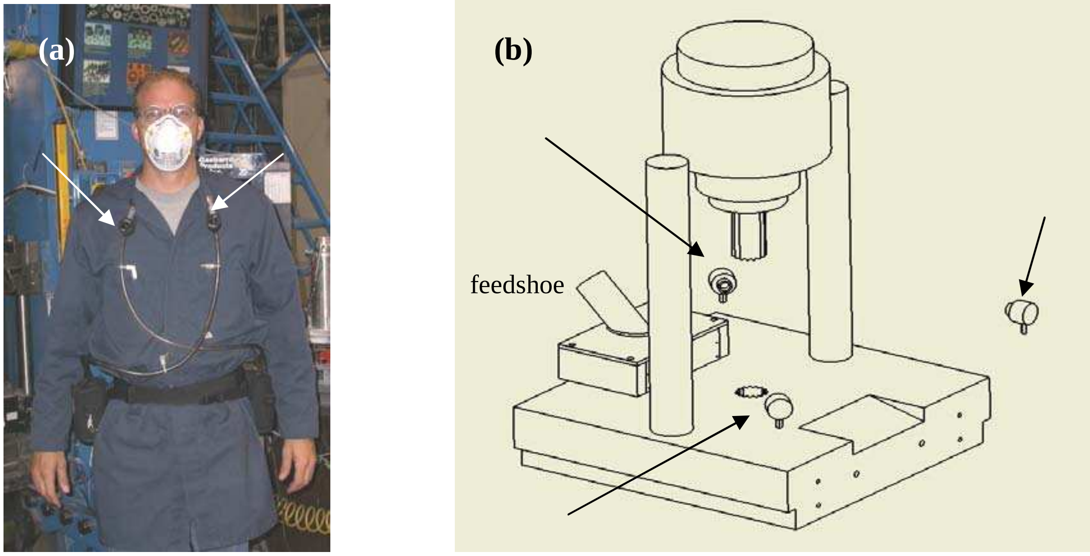

Figure 4. Locations of air samplers (a) in the breathing zone of the press operator and (b) inside the press

III. Process Monitoring

While performing the air sampling on the press operator and inside the press during the production run, the compaction process was monitored by measuring the compaction and ejection forces for each compacted part. Typical ejection curve is shown in Figure 5. The ejection stripping pressure corresponds to the force needed to start the ejection process divided by the friction area of the part in contact with the die walls. Stability of the process for the different mixes is reported in this study as a function of the scatter of the maximum compaction pressure recorded for each part, and as a function of the scatter of the part weight, done by sampling and weighting one part out of ten, for a total of about 100 parts.

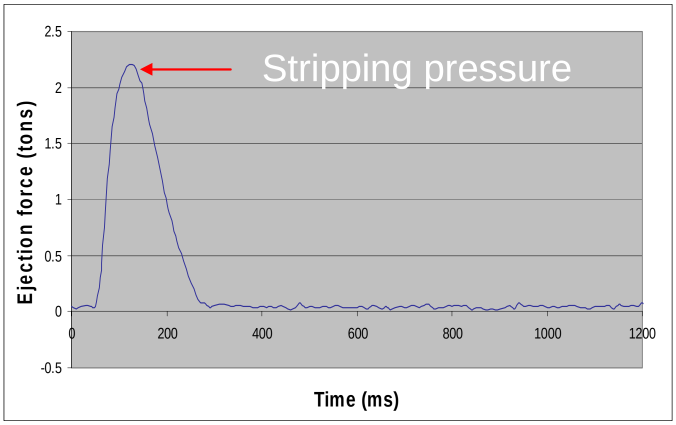

Figure 5. Typical ejection curve

RESULTS

I. Bonding Efficiency

The benefits of the diffusion and organic bonding treatments on the bonding efficiency of the copper and nickel vs. the conventional un-bonded mixing procedure were first evaluated by characterizing the dusting resistance of the four different 1.5Mo-4Ni-2Cu steel powder materials. The carbon, copper and nickel dust resistance, expressed as the percentage retained in the mix vs. the initial mix concentrations, are reported in Figure 6.

The dust resistance of *carbon*, which corresponds both to graphite and lubricant, was relatively high and similar whatever the mixes, with values between ~67% for the un-bonded CM mix, ~71% for the organic bonded OBM and OBM-F mixes and ~80% for the diffusion-bonded DBM mix. The similar conventional admixing procedure of the graphite and lubricant explains this result. In fact, ACRAWAX C lubricant is recognized as having some bonding properties when dry admixed in a conventional procedure. In the case of the diffusion-bonded DBM mix, because the copper and nickel powders were already partially bonded to the steel powder by the previous annealing treatment, the slightly higher dust resistance might be explained by the proportionally higher amount of lubricant available to bind the graphite. It is worth mentioning however that the bonding of carbon additives was not optimized here, because, as mentioned before, the prime objective of this study was to evaluate the benefits of the diffusion and organic bonding treatments on the bonding efficiency of copper and nickel. Previous results have shown that still higher carbon dusting resistance, above 90%, could be obtained when using adequate binder treatment procedures and conditions [1,8,10]. In fact, some additional work was done to confirm this with the same 1.5Mo-4Ni-2Cu steel alloy system, by organic binding not only the copper and nickel additives but also the graphite. Significant improvement of the carbon dusting resistance was obtained with values close to 93%.

CM: 1.5Mo steel + 4Ni + 2Cu + 0.6C + 0.8Wax, Conventional mix
DBM: Diffusion Bonded (1.5Mo steel + 4Ni + 2Cu) + 0.6C + 0.8Wax, Conventional mix
OBM: Binder treated (1.5Mo steel + 4Ni + 2Cu) + 0.6C + 0.7Wax, Conventional mix
OBM-F: Binder treated (1.5Mo steel + 4Ni fine+ 2Cu) + 0.6C + 0.7Wax, Conventional mix

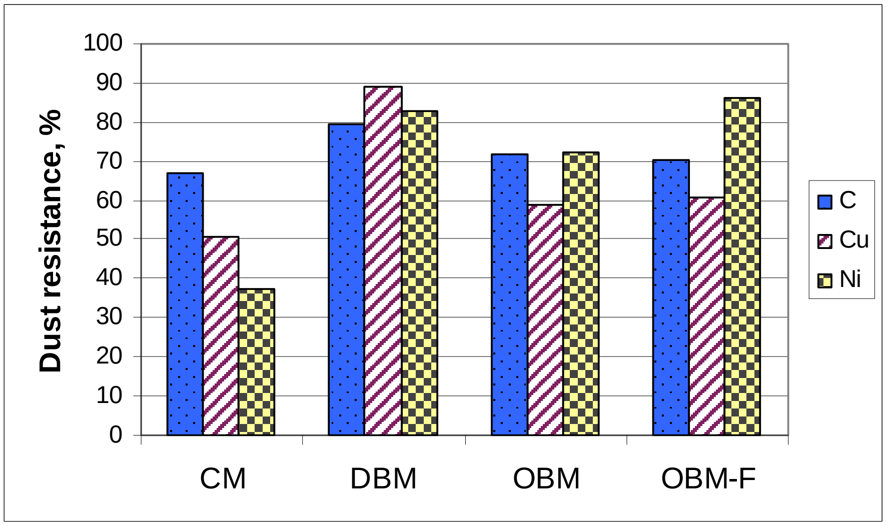

Figure 6. Carbon, Ni and Cu dust resistance of the four various 1.5Mo-4Ni-2Cu powder mixes

In regards to the dust resistance of *copper*, the binder-treated mixes show a ~20% improvement vs. the conventional un-bonded mix, with values respectively of ~60% vs. ~50%. Nevertheless, a significant higher copper dust resistance was still observed with the diffusion-bonded DBM mix, close to 90%, showing the benefits of partially diffusing this additive to the coarse steel powder to increase the bonding strength. It is interesting to note that for similar alloy systems, much lower dust resistance, close to 25% for an un-bonded powder mixes is reported in another study [1]. Much higher Cu dust resistance, ~ 50-60%, is reported in the same study for organic-bonded powders. Difference in Cu dust resistance for the conventional un-bonded mix between this study and the previous one can be explained by the fact that different grades of Cu were used. The use of a much finer copper powder in the previous study explains the lower dust resistance of the un-bonded mix. It is interesting to note that the binder treatment was able to bind this finer copper powder to the steel powder and reach similar dust resistance as those achieved in this study with a coarser Cu grade. Still higher dust resistance (~ 90%) may be reached when using coarser copper powder, such as typical –200 mesh grade available in the market [8]. Nevertheless, the size of copper affects the size of pores left behind when Cu melts and may affect the part homogeneity and dimensional consistency. It is therefore crucial to select adequately the size of copper to optimize both the copper distribution and bonding.

Looking now to the bonding of *nickel*, the lowest dusting resistance values were obtained with the conventional mix CM, with a value close to 37%. Again, the low particle size of the nickel additive (D50~8µm, for Ni 123) in an un-bonded powder mix explains this result. The nickel dust resistance was significantly improved when the binder treatment was performed, increasing by about ~2 times and reaching values of 72% for OBM. Still higher dust resistances were obtained for the diffusion-bonded DBM mix, and the organic bonded mix OBM-F prepared with the extra fine nickel powder T110 (D50~1.5µm), with values respectively of 83% and 86%. As for copper, the partial diffusion of the nickel additive to the coarser steel powder explains the high bonding strength observed for the diffusion-bonded mix DBM. If the use of fine particle additive is detrimental to the dusting of un-bonded powder mixes, the use of extra fines additives (Ni T110, D50~1.5µm) combined with an adequate binder treatment results in more efficient bonding, sufficiently strong to withstand high turbulence flow in the elutriation test. The reduced dusting tendency of extra fine nickel powder was already observed previously by Azzi et al [10]. In fact, with steel alloy containing slightly lower amount of alloying additives (0.5Mo-1.75Ni-1.5Cu steel alloy), the nickel dust resistance rose up to 97% for a binder-treated mix containing this extra fine nickel grade. It is worth mentioning that the vey high Ni dust resistance achieved with both organic-bonded treated mixes confirmed that the bonding strength is high enough to withstand subsequent mixing.

Figure 7 shows SEM micrographs for all the powder mixes, as well as the corresponding micrographs obtained by mapping the Fe, Cu and Ni elements with a RX spectrometer. Even though the mapping is preferably done on planar surfaces, it was positively used here with a higher depth-of-field to help the identification of the alloying additive particles. The efficient bonding of the nickel and copper is clearly seen for the diffusion and organic bonded mixes, as compared to the un-bonded conventional mix where the copper and nickel are mainly free and not attached to the iron particles. Dark zones in the backscattered SEM images correspond to the graphite and lubricant. More precisely, in the diffusion bonded DBM mix, the nickel particles are well dispersed and bonded to the iron powder by metallurgical bonds. In the organic bonded mixes, the nickel particles as well as the finest copper particles are located and bonded in the anfractuosities of the coarser iron particles. It appears that the capacity of a particule to be efficiently bonded by the binder-treatment within the asperity of the iron particules increased as its size is reduced. The location of these fine particules is likely influenced by their higher attraction to the surface of the iron particules due to the Van der Walls forces. Less copper powders are seen on the micrographs because of their higher particle size distribution.

II. Air sampling Results in a P/M Production Environment

The concentration in mg/m^3 of metallic and inhalable dusts that were collected on the press operator and inside the press close to the compaction area of the industrial mechanical press after the compaction of approximately 1000 parts is presented in Figure 8. Similar scales were used for the copper, iron and nickel dust concentrations to be able to compare easily the level of metallic dusts collected. Before interpreting the results, it is worth remembering that the OSHA Occupational Exposure Limits (OELs) for each of these metallic particulates are respectively 1, 1 and 10 mg/ m^3 for copper, nickel and iron respectively. However, OEL values down to 0.5 mg/m^3 for nickel are used in some countries, mainly within the European Union. Therefore, a limit of 0.5mg/m^3 is used in this text since it is a more restrictive value to assess the risks for workers.

For *copper*, even though the dusting resistance value of the different powder mixes were not higher than 60%, it is clearly seen that the concentration of copper dust is very low, in fact below 10% of the OEL value for both the dust collected on the press operator and inside the press. The particle size of the two different copper powders used having in fact a D50 close to 15 and 45 µm, this result shows that, even if the dusting resistance was not so high, mainly due to the use of high turbulent conditions during the test, it is sufficient to avoid significant copper dusting in the production working area. This clearly shows also that the conditions of the tests fluidize much more particles than what is observed under normal compaction conditions.

For *iron*, the concentration in the air is slightly higher than copper, between 0.1 and 0.2 mg/ m^3 for each of the powder mixes, both for the press operator and inside the press near the compacting area, which remains quite low as compared to the OSHA OEL of 10 mg/ m^3. The slight differences obtained between each mix and even between the sampling location is believed to be within the error margin for the sampling method used considering the fact that iron is the major constituent of the powder mixes (>90%wt).

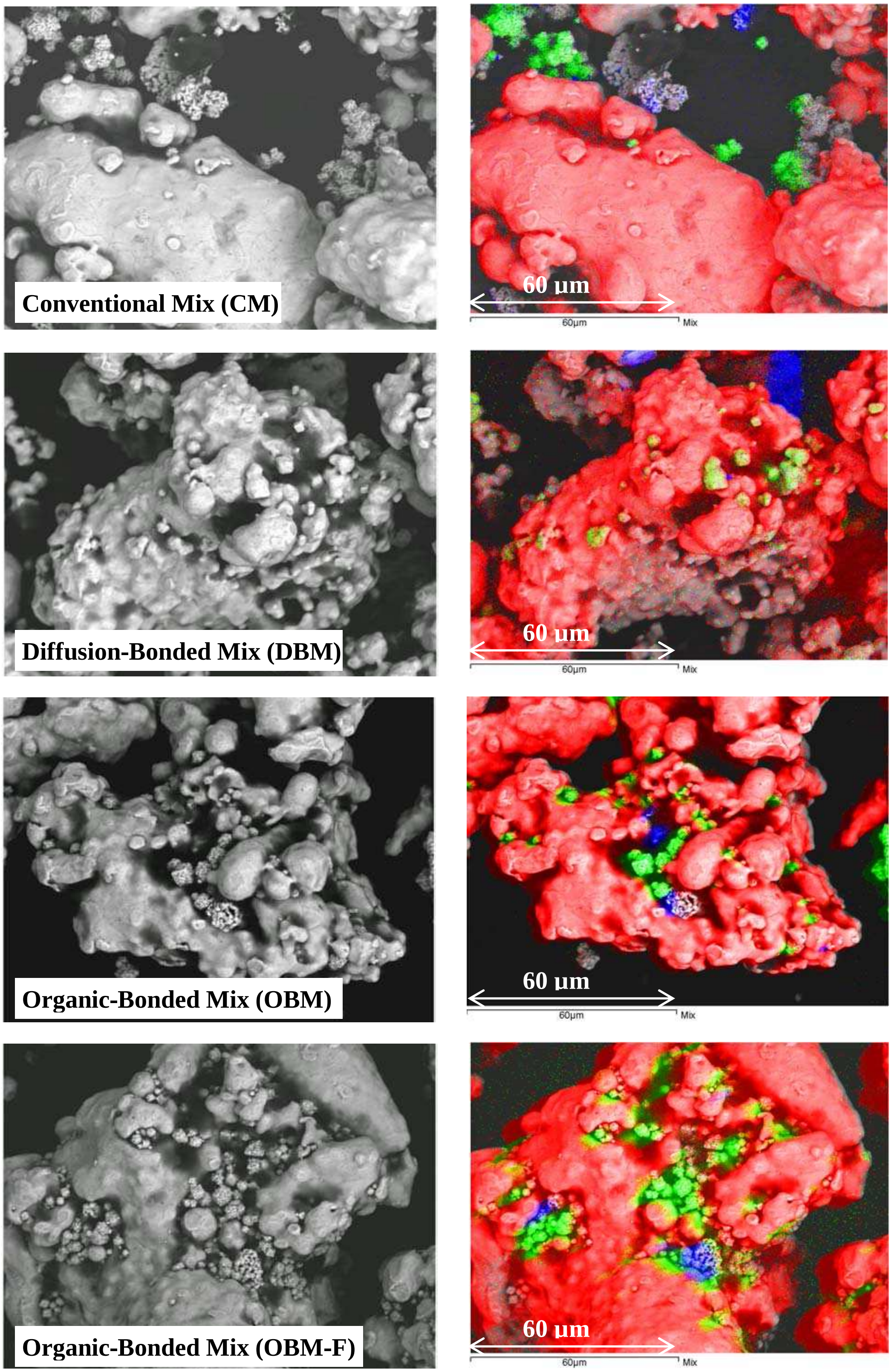

Figure 7. SEM micrographs (1000X) and RX mapping of Fe (red), Ni (green), Cu (blue) of the four powder mixes

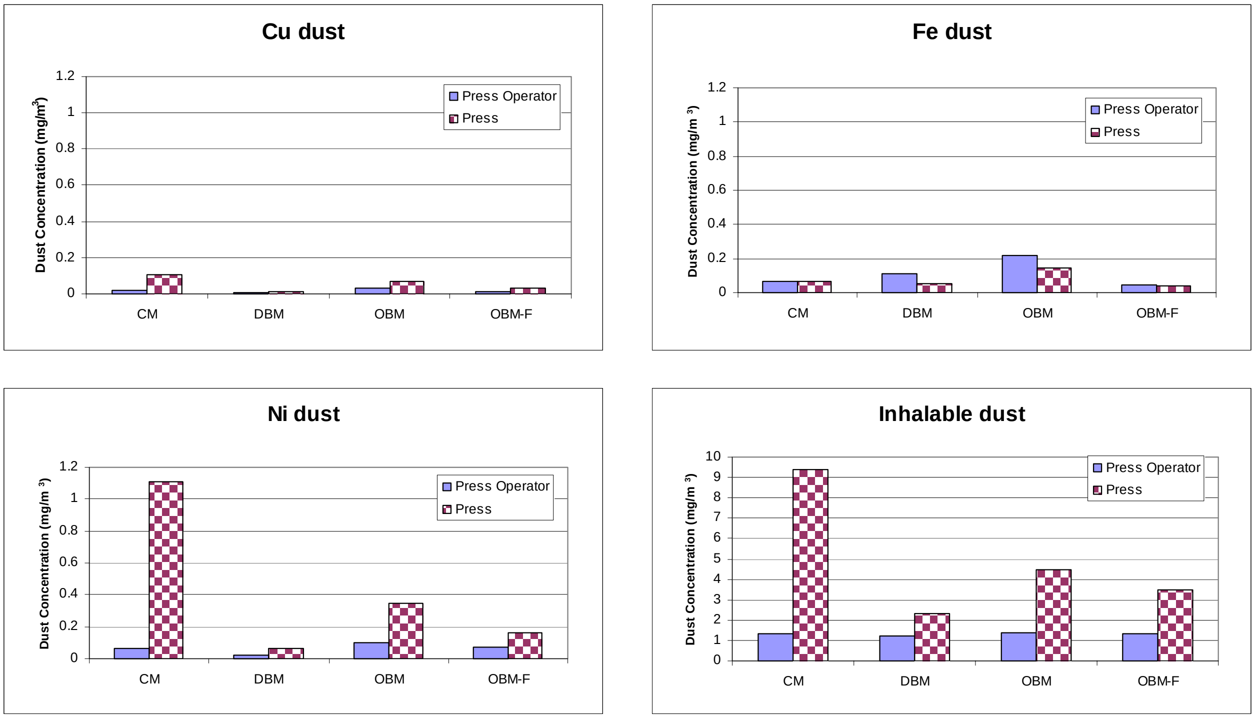

Figure 8. Cu, Fe, Ni and Inhalable dust concentrations recorded in the breathing zone of the press operator and inside the press close to the feeding zone.

For *nickel*, very different results were achieved depending on the location of the sampler. In fact, in the conditions of the exposure scenario used, the concentration of nickel in the atmosphere collected on the press operator is well below the OEL of 0.5 mg/m³, mainly below 0.1 mg/ m^3 for all the powder mixes used. However, completely different behavior is obtained regarding the nickel concentration in the dust collected inside the press close to the compaction and feeding areas. A value higher than 1 mg/ m^3 was obtained for the un-bonded regular mix CM, which is higher than the OEL value of 0.5 mg/ m^3 used in some European countries and even higher than the OSHA OEL of 1 mg/ m^3. The significantly lower particle size of the nickel combined with its greater concentration in the mix as compared to the copper powder likely explains these results. On the other hand, the bonding efficiency of nickel is clearly seen in the diffusion-bonded powder mix DBM that shows the lowest value of nickel concentration in the air inside the press, below 0.1 mg/ m^3. The benefit of bonding the nickel powder to the coarser steel powder was also seen for the organic-bonded treated mixes. Concentrations of nickel captured were respectively 0.36 and 0.16 mg/ m^3 for OBM and OBM-F mixes, which remain lower than the OEL of 0.5 mg/ m^3 and is approximately 3 to 7 times lower than the Ni dust captured with the conventional un-bonded mix. The nickel dust collected with the organic-bonded mixes, mainly the one made with fine Ni 123, remains higher than that achieved with the diffusion-alloyed mix. The excellent bonding of the extra fine nickel powder as reported earlier [10] is confirmed with these results.

Finally, the inhalable dust, that includes the copper, nickel and iron dusts, but also the graphite and lubricant dusts were quite stable whatever the powder mixes for the air collected close to the breathing zone of the press operator, with values close to 1 mg/ m^3. Considering the relatively low concentration of graphite and lubricant compared to nickel, copper and iron, it is a bit surprising at a first glance that the total inhalable dust is 5 to 10 times higher than the amount of all the metallic dust collected. However, if we consider the very fine particle size and low specific gravity of graphite and lubricant (respectively of 2.3 and 1 g/cm^3) and we estimate the number of particles within the mix instead of the weight portion, it is not a surprise to see that most of the dust created during compaction is constituted of graphite and organic lubricant. Looking to the inhalable dusts collected inside the press, significantly higher values were obtained, and in particular for the un-bonded mix CM that reached 9 mg/ m^3 while 2.3 mg/ m^3, 4.5 mg/ m^3 and 3.5 mg/ m^3 were respectively measured for the diffusion-bonded mix DBM and the binder-treated mixes OBM and OBM-F. The fact that the graphite and lubricant were never bonded in these powder mixes, but only admixed to the pre-mixes, may explain partly these results, as shown previously with the dust resistances of carbon that were close to 60%. It is worth mentioning that by performing the binder treatment after admixing the graphite with the nickel and copper to the steel powder, significantly higher dust resistance of carbon of 90-95% could be reached [8] that would lead to lower inhalable dust values.

III. Compaction and Ejection Behavior

The mean compacting pressures and ejection stripping pressures as recorded during the production runs are given in Figure 9. The compressibility of the two organic-bonded powder mixes OBM and OBM-F was significantly better than that of its diffusion-bonded counterpart DBM, the difference being typically of about 70-80 MPa (5-6 tsi) for a green density of 7.0 g/cm³. The higher compacting pressure required to reach 7.0 g/cm³ with the DBM powder is due to the partial diffusion of Ni and Cu in the iron grains during annealing. On the other hand, the compressibility of the two organic-bonded powder mixes OBM and OBM-F was equivalent or slightly better than that of the conventional mix CM, indicating that the organic-bonding process is not detrimental to the compressibility of base powder. The Figure 9 shows also that the organic-bonded powder mixes gave the best ejection performance amongst all the materials tested, with ejection stripping pressures lower than about 20% and 10% as compared respectively to the diffusion-bonded and the conventional un-bonded powder mixes. These results confirmed the very good lubricating properties of the binder used to bond the Ni and Cu as reported in previous studies by St-Laurent et al [1,8].

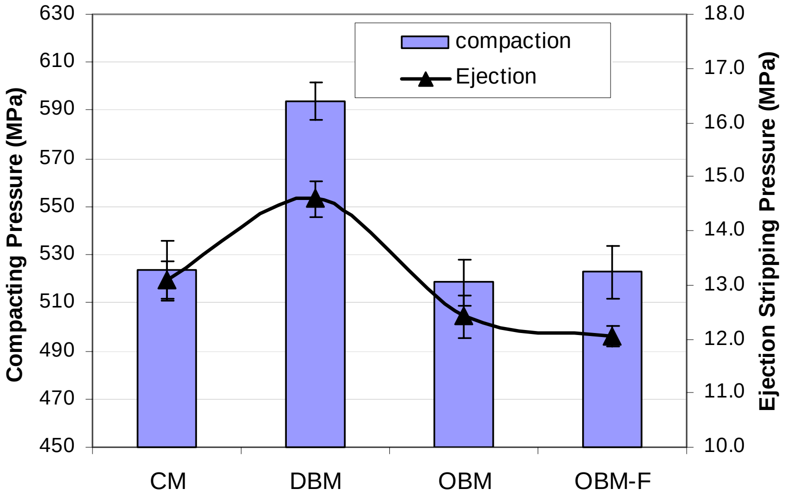

Figure 9. Mean values of compaction pressures and ejection stripping pressures for the four series of about 1000 sprockets compacted at 7.0 g/cm³ on the industrial mechanical press.

The stability of the compaction process for the different mixes is illustrated by the scatter of the compaction pressure (Figure 10) and the part weight (Figure 11) during each run. Best results were obtained with the diffusion-bonded powder mix DBM and the organic bonded powder mix OBM. When extra fine nickel T110 is used (OBM-F), slightly higher scatter was observed, while still higher scatter was observed with the conventional un-bonded powder mix CM. Indeed, the standard deviation for the maximum compaction pressures was 7.8 MPa for DBM, 9.4 MPa for OBM, 11.2 MPa for OBM-F and

13.2 MPa for CM. This represents an improvement of 29% when using the organic bonded powder mix OBM instead of the conventional un-bonded mix CM. Similar trend was obtained with the standard deviation of part weight as shown in Figure 11. Indeed, a reduction of 26% and 49% in part weight variation was observed when using respectively the organic bonding treatment (OBM) and the diffusion bonding treatment (DBM) as compared to the un-bonded powder mix CM. Similar scatter in part weight was obtained with the un-bonded powder mix CM and the organic bonded powder mix using the extra fine nickel additive, with standard deviation respectively of 0.52% and 0.5%. This behavior is in agreement with the flow characteristics of these powder mixes (Figure 12). Indeed, both the regular mix CM and the organic bonded mix OBM-F mix do not flow at all. It is worth mentioning that the goal of this study was not to optimize the flow behavior of these powder mixes by either adding flow additives or optimizing the binder treatment procedure. In fact, some additional work done on a mix for which the binder-treatment was optimized in order to obtain an excellent flow and achieve very good copper, nickel and graphite bonding, showed a significant improvement in the stability of the compaction process with an excellent standard deviation for part weight of 0.18% and a standard deviation in compaction pressures of 4.3 MPa.

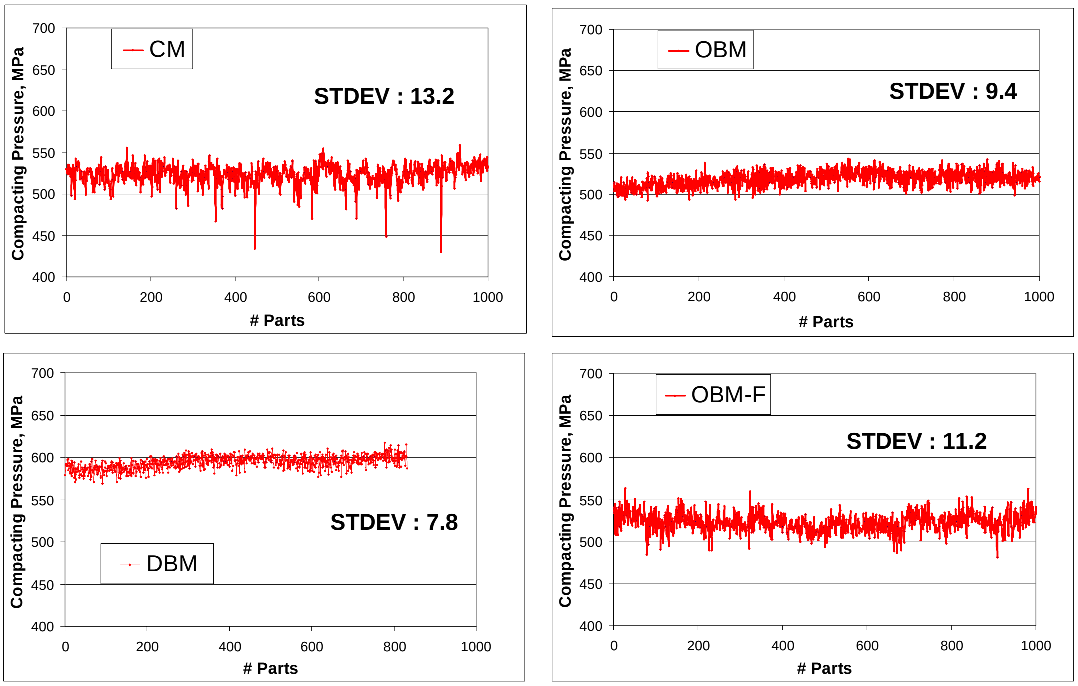

Figure 10: Scatter in compaction pressures along the compaction runs for the four powder mixes

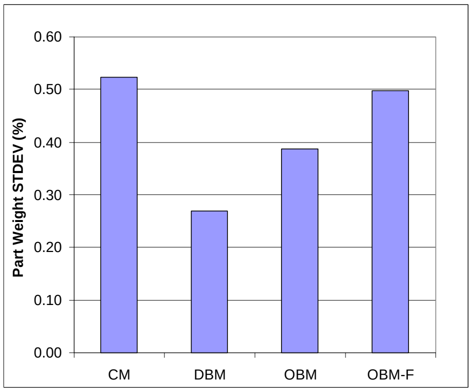

Figure 11: Part weight standard deviation

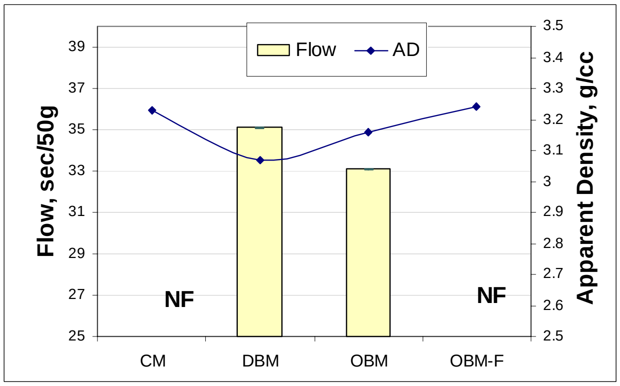

Figure 12: Flow and Apparent density of the mixes studied

CONCLUSIONS

In this paper, the bonding efficiency of binder-treated 1.5Mo-4Ni-2Cu powders was evaluated on an industrial press and compared to that of regular un-bonded and diffusion-bonded powders. An air monitoring strategy was used to evaluate both the worker exposure to dust, as well as the dust generated close to the compaction area inside the press. The bonding efficiency was also evaluated on the different powder mixes through the determination of the dusting resistances of the carbon, copper and nickel using an elutriation method. The main conclusions are:

- Even though the elutriation test is performed with a strong air flow to fluidize the particles and evaluate the dusting resistance of these particles, this study clearly shows the benefits of performing air monitoring directly close to the compaction press during the production of a series of parts. Indeed, if the elutriation test lead to higher dusting resistance of nickel as compared to copper, the concentration of nickel in the workplace air was higher than the concentration of copper, mainly due to differences in particle size distributions.

- The level of dust, mainly for nickel, varied significantly depending on the location of the air sampler. Indeed, even though the concentrations of nickel measured in this study on the press operator were low (<0.1 mg/ m^3), the much higher concentrations measured close to the pressing area, especially for a *conventional un-bonded mix* (>1 mg/ m^3) should be treated cautiously. Thus, *for un-bonded mixes*, it is recommended to assess the risk for the worker by performing personal air sampling in the working area
    - following a statistical plan to take into account the errors associated with such a sampling method,
    - for a longer period of time up to 8 hours to take into account the air fluctuations that may occur in the workplace.

- This study showed for steel powder mixes containing a high amount of alloying elements the clear benefit of using:
    - *diffusion-alloyed* powders for which nickel and copper are partially diffused to steel particles,
    - or *organic-bonded* powders, for which the nickel and copper are bonded to the steel particles using a polymeric binder and a special bonding process such as the FLOMET™ process.

  This will translate in a better working environment, and could potentially eliminate significant capital investment to improve the air quality in the workplace.

ACKNOWLEDGEMENTS

The authors would like to acknowledge Patrick England from IMI-NRC that performed the compaction and air sampling tests, Daniel Simard from IMI-NRC that performed the SEM and RX analyses and Benoît Chartrand from QMP for his help in evaluating the air sampling strategy and for the fruitful discussions on the results of these tests.

REFERENCE

---

1 St-Laurent, S. and Gélinas, C., "High Performing Mo-Ni-Cu Bonded Powders for Medium to High Density PM Applications", EuroPM2007, International Powder metallurgy Congress & Exhibition, Toulouse, France; 15-17 October 2007.

---

2 Bailey, S., "REACH - will “derived no-effect levels” (DNELS) set under REACH replace occupational exposure limits?", Newsletter of the British Occupational Hygiene Society, Feb. 2007, Vol. 20, Issue 1, pp.13-14.

3 United States National Institute for Occupational safety and Health (NIOSH), Method 7300, issue 3, March 15, 2003 in NIOSH Manual of analytical methods, fourth edition, http://www.cdc.gov/niosh/nmam/pdfs/7300.pdf

4 United States Occupational Safety and Health Administration (OSHA). Method ID-125G: Metal and metalloid particulates in workplace atmospheres (ICP analysis), http://www.osha.gov/dts/sltc/methods/inorganic/id125g/id125g.pdf

5 Health & Safety Executive, Methods for the determination of hazardous Substances Nickel and inorganic compounds of nickel in air (except nickel carbonyl) : Laboratory method using flame atomic absorption spectrometry or electrothermal atomic absorption spectrometry. MDHS 42/2 , march 1996, ISBN 0-7176-094-2.

6 Gosselin, F., "Segregation-Free Metallurgical Powder Blends Using Polyinylpyrrolidone Binder", U.S. Patent N°5,069,714.

7 Champagne, B., Cole, K. and Pelletier, S., "Segregation-Free Metallurgical Blends Containing A Modified PVP Binder", U.S. Patent N°5,432,223.

8 St-Laurent, S., Gélinas, C., Thomas, Y. and Azzi L., "Using Binder-Treatment Technology for High Performance Steel Powder Mixes", PM2TEC 2005: 2005 International Conference on Powder Metallurgy & Particulate Materials; Montreal, Quebec; Canada; 19-23 June 2005, pp. 3.110-3.123.

9 Technical Info on SKC website: “Operating Instructions Airlite ® sample pump catalog No. 110-100”, http://www.skcinc.com/instructions/37814.pdf, and “IOM sampler, A gold Standard for Personal Inhalable Particulate Sampling”, http://www.skcinc.com/instructions/1050.pdf

10 Azzi, L., Pelletier, S., Stephenson, T. and St-Laurent, S., "Effect of Nickel Type on Properties of Binder-Treated Blends", PM2TEC 2005: 2005 International Conference on Powder Metallurgy & Particulate Materials; Montreal, Quebec; Canada; 19-23 June 2005, pp. 10.1-10.14.