

# **INFLUENCE OF THE PARTICLE SIZE DISTRIBUTION AND MORPHOLOGY OF NI AND CU POWDERS ON THE DIMENSIONAL CHANGE OF SINTERED PARTS**

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## **ABSTRACT**

Dimensional change is one of the most important properties of PM structural parts and part producers are demanding tight tolerances. Copper and nickel elemental powders are commonly used as admixed additives to tailor targeted properties but both result in a significant dimensional change. Both elements generally increase the mechanical properties but copper is known to promote growth while the use of nickel leads to shrinkage.

This paper focuses on the impact of characteristics of copper and nickel additives on the properties of the resulting PM parts and particularly on dimensional change. The particle size distribution (PSD) and the morphology of different types of Cu and Ni additives were characterized and the link between these characteristics and the dimensional change was investigated under slow cooling rates.

## **INTRODUCTION**

As powder metallurgy expands to the production of high-performance components with tight tolerances and superior mechanical properties, it becomes of critical importance to better predict and control the dimensional change of sintered parts since secondary operations like sizing and machining are generally costly and difficult to perform. Copper and nickel are commonly used as alloying elements in PM steels. Copper is generally added as an elemental powder to premixes while nickel is suitable to be pre-alloyed or premixed. PM materials containing a combination of these alloying elements are commonly used for high-performance application (e.g. FLNC-4405).

During typical sintering cycles, admixed copper melts at around 1083°C and is redistributed throughout the steel compact by liquid phase displacement, grain boundary diffusion and ultimately by volume diffusion in the iron lattice. The redistribution mechanisms of copper during sintering lead to swelling [1, 2, 3]. The presence of carbon in the Fe-C-Cu system tends to decrease the swelling mainly because carbon decreases the wettability of liquid copper which has a direct impact on the spreading efficiency of the liquid phase and, hence, grain boundary penetration [3]. Elemental copper powders commonly used in PM

have an average particle size of 50-70  $\mu\text{m}$ . Previous studies showed that the size of copper particles has a significant effect on the extent of the swelling of PM parts [2, 4]. This amplitude of the swelling seems to vary considerably with, among other things, the nature of the base powder, the type of formulation and the sintering profile characteristics.

On the other hand, fine nickel particles remain fully solid at typical sintering temperatures. Carbonyl nickel powder with  $d_{50}$  around 7-10  $\mu\text{m}$  is the most commonly used nickel additive in PM steels. Different mechanisms responsible for the redistribution of nickel during sintering have been described in numerous publications but do not seem to be unanimous [1, 5, 6, 7, 8]. It seems that volume diffusion of nickel in iron is limited, so that surface and grain boundary diffusion mechanisms are dominant [7, 8]. Hence, it is not unusual to witness the presence of nickel rich areas in sintered parts, caused by the slow redistribution rate of the nickel in steel particles [6, 7, 8]. In terms of dimensional change, the presence of fine nickel particles increases the total specific surface area of the compact causing larger shrinkage at holding temperature [1]. In that sense, the particle size distribution of nickel has a direct effect on the dimensional change of PM steels.

The dimensional change response of PM steels containing both admixed copper and nickel is not straightforward. It has been demonstrated that there is an interaction between both elements during the course of sintering [4, 5, 9, 10]. This interaction has a significant impact on the dimensional response and depends on the sintering profile characteristics (heating rate, sintering temperature and time, etc). Nabeel et al. suggests that the formation of Cu-Ni liquid phase, which has a lower wetting angle than liquid copper, enhances the redistribution of both species in the compact [5]. In another study, Lindsley et al. found that the use of fine copper with nickel diminishes the extent of growth in FLNC-4408 PM steels when compared with regular copper [4]. This is also in line with Strobl et al. who used copper coated iron particles in their work, an extreme case which can be considered as infinitely fine copper particles, and found that swelling was greatly reduced or even suppressed [11].

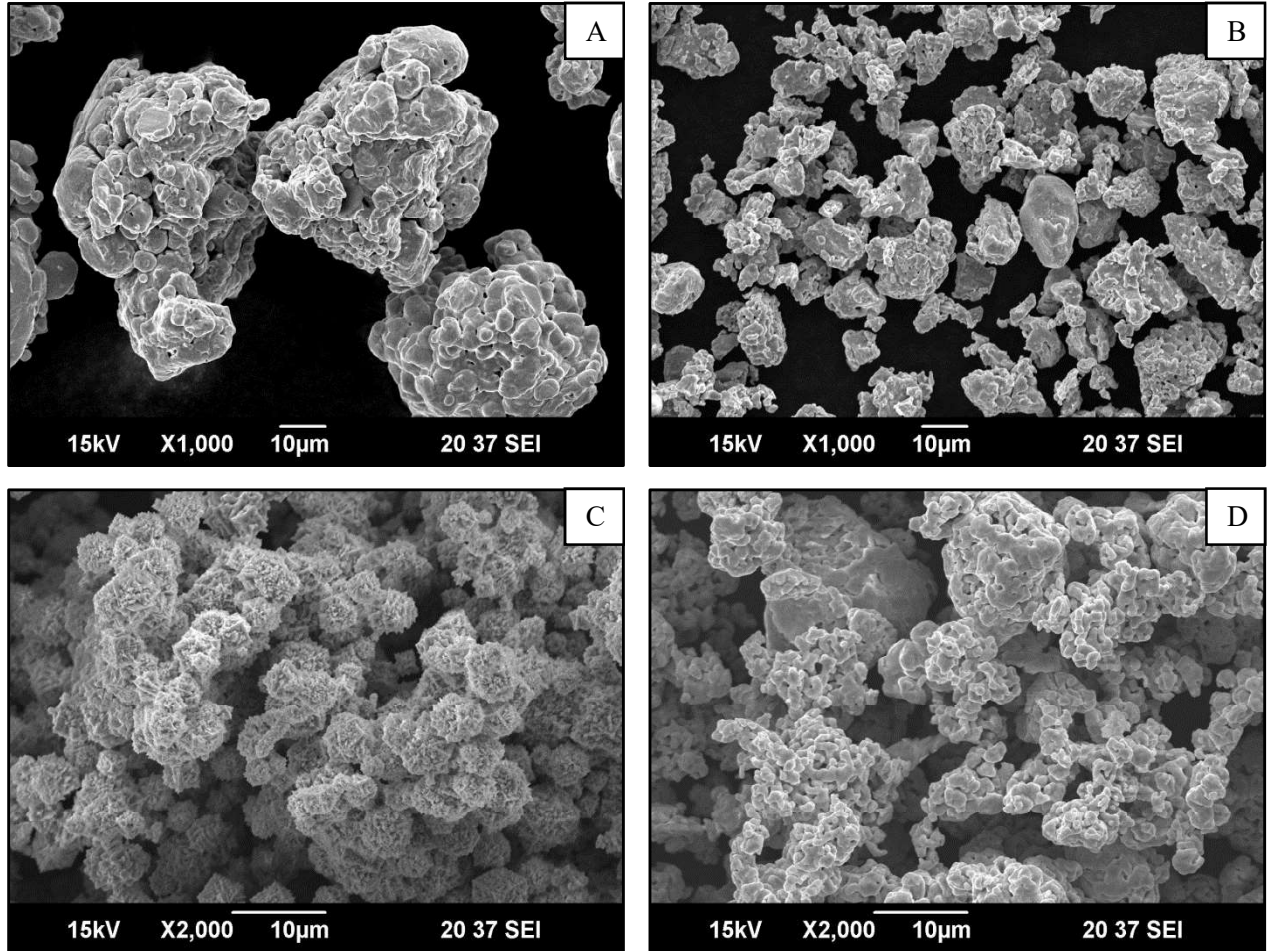
Dilatometry is a powerful characterization method to appreciate the *in-situ* dimensional change of a sample during sintering cycle [12, 13, 14]. Dilatometry provides useful information on thermal expansion, phase transformations, swelling and densification.

In this work, a dilatometry study was carried out by the authors to evaluate the dimensional response of PM steels containing elemental copper and nickel additives. The main objective of this study was to evaluate the effect of specific characteristics of copper and nickel additives on the sintering kinetics, particularly the particle size distribution of copper additives and the morphology of nickel particles. The dilatometry results shown in this study contribute to a better understanding of the interaction between copper and nickel during sintering.

## **EXPERIMENTAL PROCEDURE**

A commercially available water-atomized ferrous powder, pre-alloyed with 0.85% Mo (ATOMET 4401), was used in this study. All mixes contained 0.60% TIMREX<sup>®</sup> KS15 synthetic graphite (Timcal). Two elemental copper powders, both produced by water-atomization and sieving, but having different particle size distributions, were selected: regular copper (RegCu) and fine copper (FineCu), see Table 1. It can be appreciated that the morphology of copper particles is qualitatively similar for both copper powders (Figure 1). Two types of nickel with different particle morphologies were used: a nickel powder produced

by a carbonyl process (CarbNi) and a nickel produced via a reduction, milling and sieving process (ReduNi). As seen in Figure 1, the production process had a significant impact on the morphology of the nickel particles. The surfaces of particles produced by the carbonyl process have a spiky appearance which is typical of this type of production process, while particle surface looks smoother in the case of reduced nickel.



**Figure 1 : SEM images of powder additives. (A) Regular copper. (B) Fine copper. (C) Carbonyl nickel. (D) Reduced nickel.**

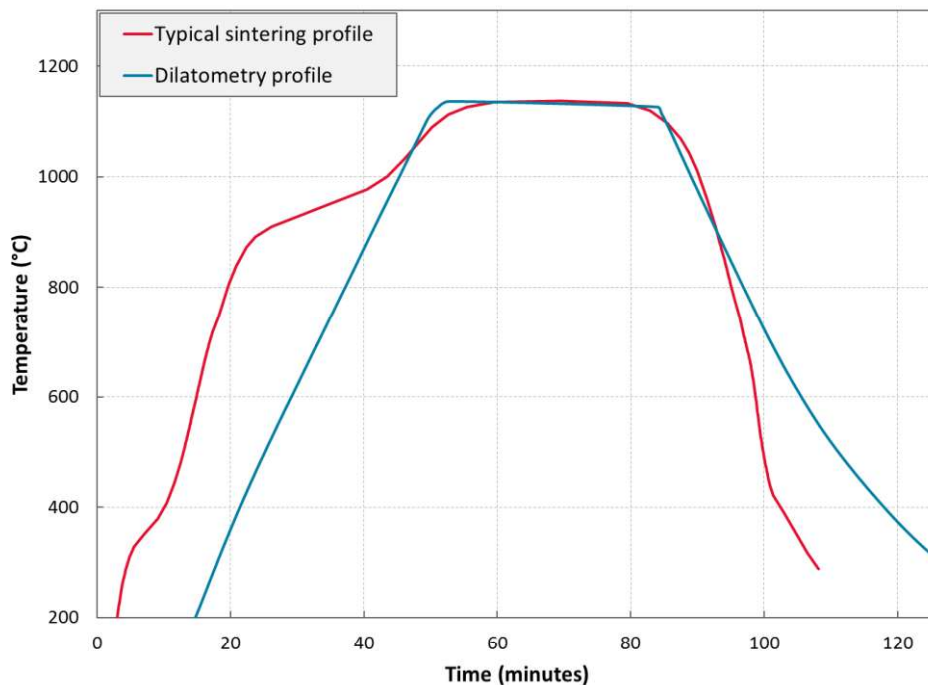
The particle size characteristics of the different additives were determined by laser diffraction using a HELOS particle size analyzer (SYMPATEC) and are listed in Table 1. All the additives used in this study, including copper and nickel, are commercially available products.

**Table 1: Particle size properties of copper and nickel additives used**

Cu/Ni additives	d <sub>10</sub> (µm)	d <sub>50</sub> (µm)	d <sub>90</sub> (µm)	d <sub>99</sub> (µm)
RegCu	21.6	54.2	104.5	150.0
FineCu	5.7	11.7	20.3	28.2
CarbNi	1.7	7.0	22.9	55.8
ReduNi	2.7	10.4	21.9	34.8

Mixes were prepared with various amounts of the different types of copper (0% and 2%) and nickel (0% and 2%) without any lubricant in order to be used for dilatometry. Dilatometry specimens (20.00 mm x 6.35 mm x 6.35 mm) were compacted at 6.80 g/cm<sup>3</sup> using die-wall lubrication (manually applied zinc-stearate spray).

Thermal expansion was examined by a vertical push-rod dilatometer (Linseis L70) in an atmosphere composed of 90 vol% of nitrogen and 10 vol% of hydrogen. The sintering profile used for dilatometry consisted of a heating ramp of 25°C/min until 1100°C. The heating rate was slowed down to 10°C/min from 1100°C to 1120°C, and 5°C/min from 1120°C to 1130°C. Temperature was held at 1130°C for 30 minutes. The actual temperature overshoot to 1138°C at the beginning of the plateau and progressively settled down to the set-point temperature. Cooling rate was set to 20°C/min from 1130°C to room temperature. The dilatometer failed to maintain this cooling rate below 650°C. The effective cooling rate observed in the dilatometer between 650°C and 400°C is approximately 17°C/min. Prior to the sintering cycle, samples were held in the dilatometer at 125°C for 30 minutes to stabilize the system. The temperature profiles used for dilatometry trials are illustrated in Figure 2. As a comparison, a typical sintering profile without accelerated cooling is also shown in the same figure.



**Figure 2: Temperature profiles used for sintering and dilatometry**

The dilatometry profiles presented in this paper illustrate the percentage of variation of the specimen expansion during the sintering cycle, as compared with the measured length of the initial green specimen at room temperature. Every dilatometry profile was repeated twice to validate the consistency of the measurements. Sintered specimens were cross-sectioned, mounted and polished according to standard practices and microstructural characteristics were revealed using Nital (2%). All the micrographs shown in this paper were obtained using an optical microscope (Olympus GX71).

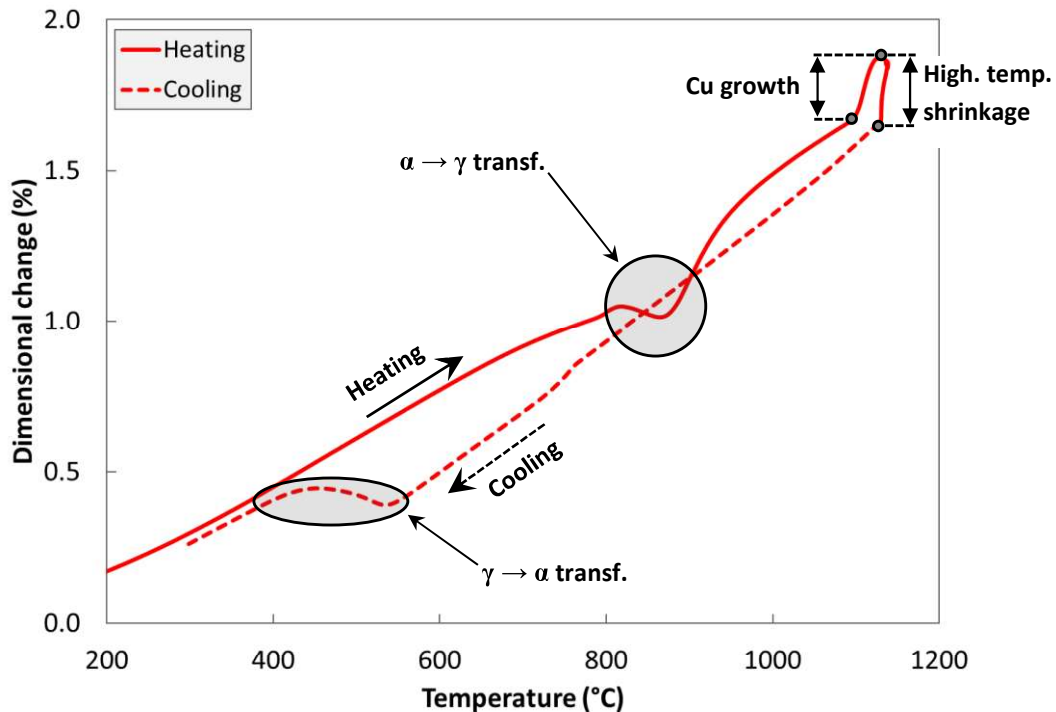
## RESULTS AND DISCUSSION

From each dilatometry profile, it was possible to extract dimensional variations associated with specific events happening during the sintering cycle. More particularly, the amplitude and the rate of the growth caused by copper as well as the shrinkages observed at sintering temperature were evaluated in each case and are listed in Table 2.

**Table 2: Dimensional variations related to different events during the sintering cycle**

Case	MPIF designation	%Cu	%Ni	Growth (Cu) (%)	Rate of growth (Cu) (%/°C)	High-temp. shrinkage (%)
Ref.	FL-4405	-	-	-	-	0.17
1	FLN2-4405	-	2% CarbNi	-	-	0.28
2	FLN2-4405	-	2% ReduNi	-	-	0.26
3	-	2% RegCu	-	0.32	0.007	0.10
4	-	2% FineCu	-	0.37	0.007	0.09
5	FLNC-4405	2% RegCu	2% CarbNi	0.42	0.011	0.25
6	FLNC-4405	2% FineCu	2% CarbNi	0.23	0.006	0.22

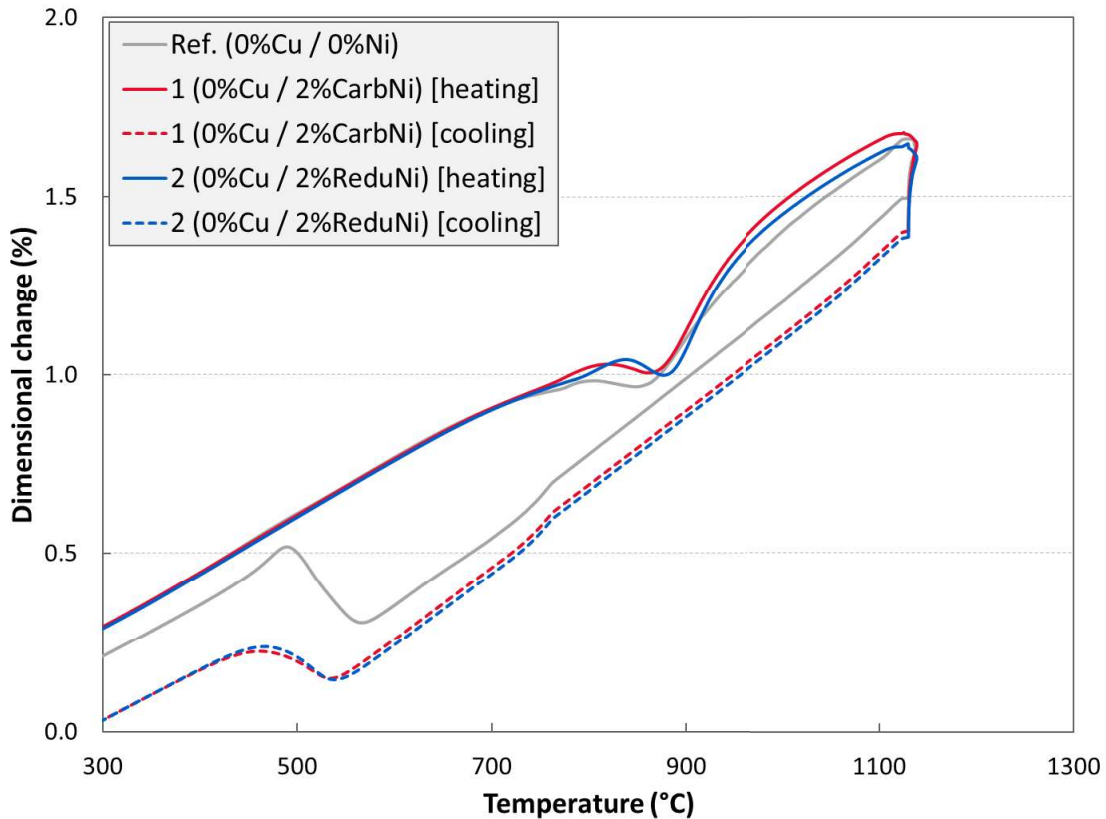
The reader can relate these values to the individual dilatometry profiles presented in the next pages. Figure 3 shows a typical dilatometry profile, illustrating the key events and their amplitudes. Shrinkages observed at sintering temperature (high-temp. shrinkage) are not perfectly vertical on the graphs due to the temperature overshoot mentioned previously. Nevertheless, the shrinkage was calculated by evaluating the difference between the maximum expansion reached at the beginning of the plateau (1130-1138°C) and the length of the specimen right before cooling began (1130°C).



**Figure 3: Typical dilatometry profile of PM steel specimen containing copper with key events.**

### Impact of the morphology of nickel particles

First, the impact of the morphology of nickel particles was studied. Figure 4 shows the dilatometry profiles of copper-free samples containing 2% of the two types of nickel (CarbNi and ReduNi). The shaded curve represents the dilatometry profile of a reference sample free of copper and nickel. Thermal expansion of ferrite during the heating phase is similar in both cases and is consistent with past studies [12, 14].



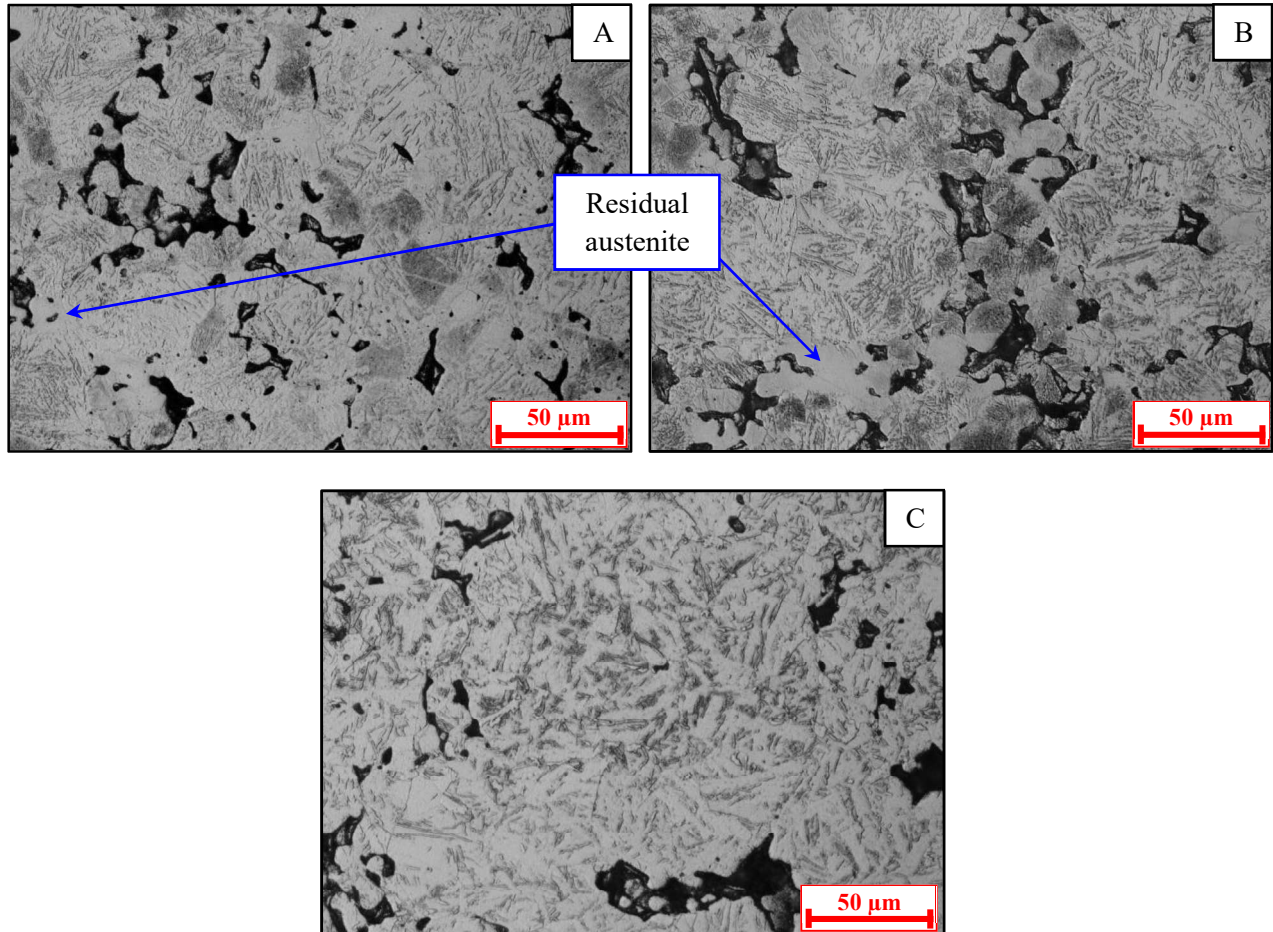
**Figure 4: Dilatometry profiles of copper-free samples containing 2% of the different nickel (FLN2-4405).**

As expected, large shrinkages are observed at holding temperature in both cases (0.26% and 0.28%). This shrinkage is larger than the reference sample (0.17%) which confirms that the presence of fine nickel particles increases the total surface energy of the specimen, resulting in more shrinkage. Nickel produced by the carbonyl process causes a slightly larger shrinkage than reduced nickel. It is difficult to correlate this tiny difference to the morphology of the nickel particles since there are also small differences in the particle size distribution of both powders. Nickel produced by the carbonyl process is slightly finer than reduced nickel, which could have contributed to the larger shrinkage observed (see Table 1).

The cooling response is also very comparable in both cases. The beginning of the transformation of austenite is delayed in time and the amplitude of the growth generated by this transformation is smaller when compared to the reference sample. This is due to the presence of residual austenite (nickel rich area) which as a higher density than equilibrium phases ( $\alpha$  and  $\text{Fe}_3\text{C}$ ), as seen in Figure 5. The final lengths of

the bars containing nickel are shorter than for the nickel-free formulation, confirming that the presence of nickel amplifies the overall shrinkage of PM steels.

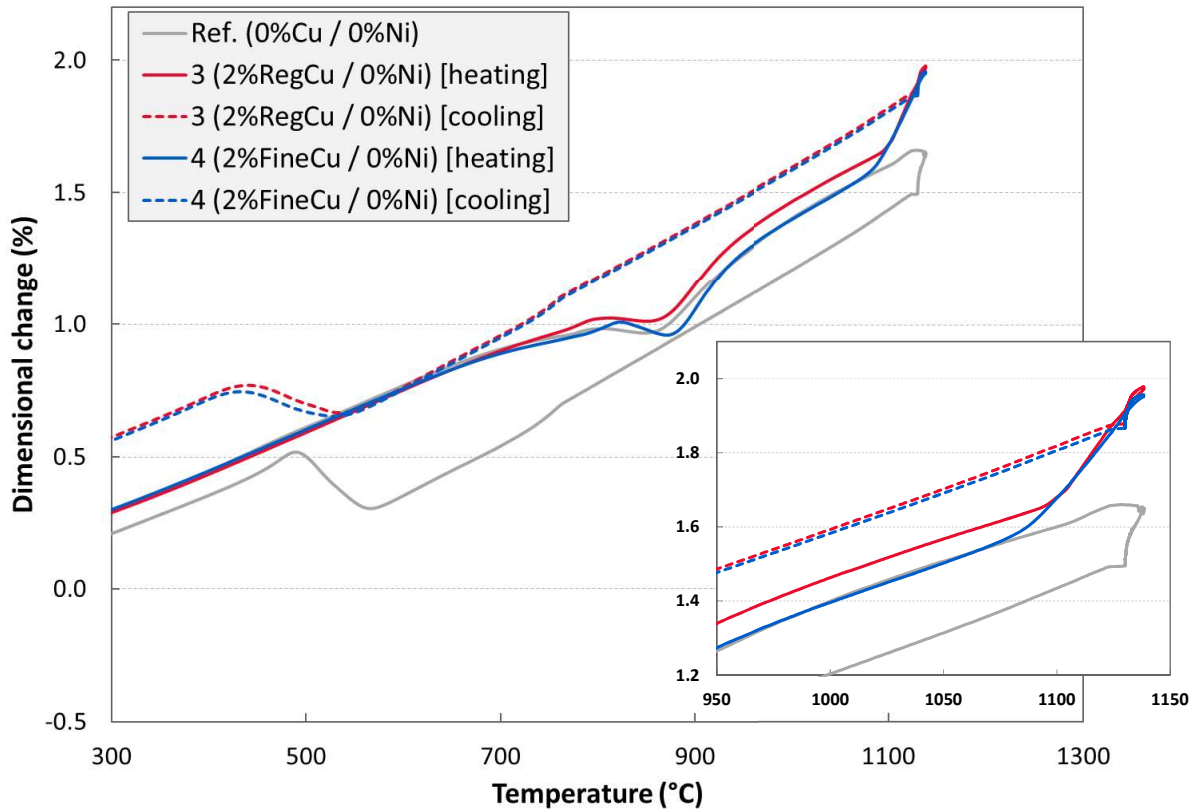
The dilatometry profiles shown here suggest that the type of nickel, and to a further extent, the morphology of the nickel particles does not have a significant influence on the overall dimensional behaviour, in the range of particle size distribution tested here.



**Figure 5 : Microstructures of sintered specimens, etched with Nital (500x): (A) 2% carbonyl nickel / 0%Cu. (B) 2% reduced nickel / 0%Cu. (C) 0%Ni / 0%Cu**

*Impact of the particle size distribution of copper additives*

The dilatometry curves of nickel free samples containing 2% copper are presented in Figure 6. Both specimens show similar expansion profiles until the  $\alpha \rightarrow \gamma$  transformation. Following this transformation, expansion is accelerated by the diffusion of graphite in the austenite phase. The two samples expand in a similar way until 1080°C where copper melts. In fact, the growth due to copper occurs earlier in the case of fine copper (~1083°C) versus regular copper (~1095°C) in the specific sintering conditions of this study. The amplitude of the growth is slightly larger in the case of fine copper (0.37% vs 0.32% for regular copper). It is important to mention that the rate of expansion during this growth appears to be nearly the same for two types of copper.

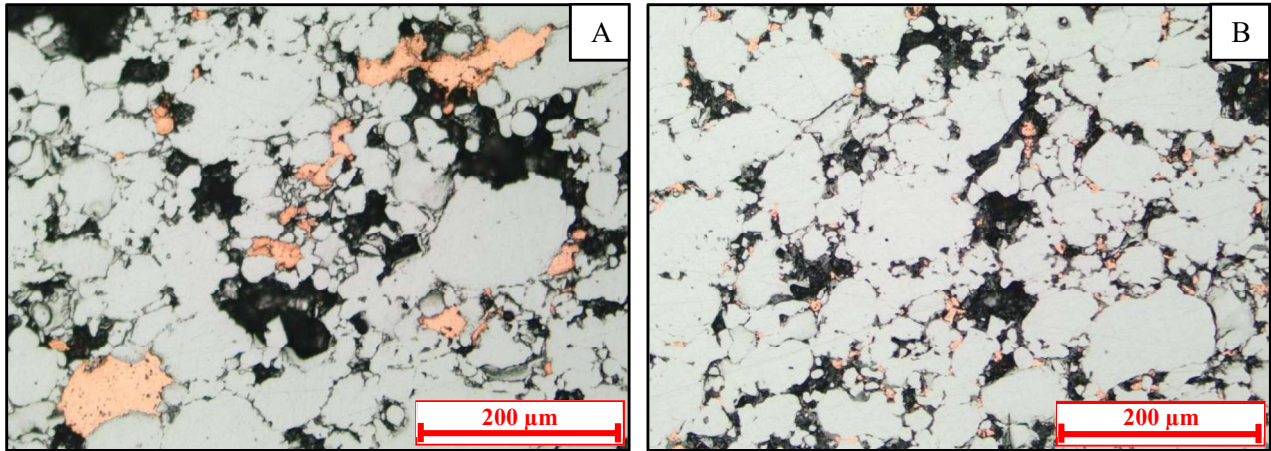


**Figure 6 : Dilatometry curves of nickel-free samples containing 2% of the different copper**

In the following paragraph, a possible sequence of events resulting in the difference observed between fine and regular copper will be suggested. Figure 7 show the initial size and distribution of copper particles in green compacts (mounted in epoxy). As a consequence of their size, fine copper particles are more homogenously distributed throughout the compact. Liquid copper has to travel smaller distances before penetrating grain boundaries and eventually copper atoms go quicker in solid solution into the iron lattice. Moreover, a better spatial distribution allows more grain boundaries to be influenced by copper diffusion, resulting in more growth. Direct volume diffusion from the copper particles into the iron lattice from the periphery of the iron particles is also enhanced by a better spatial distribution. These are some reasons that can explain the earlier and slightly larger growth seen with fine copper, during this specific sintering cycle.

In the case of regular copper, it is suggested that this growth is postponed in time since liquid copper has to travel as a liquid phase in pore channels first before generating swelling by penetrating grain boundaries or diffusing in volume.



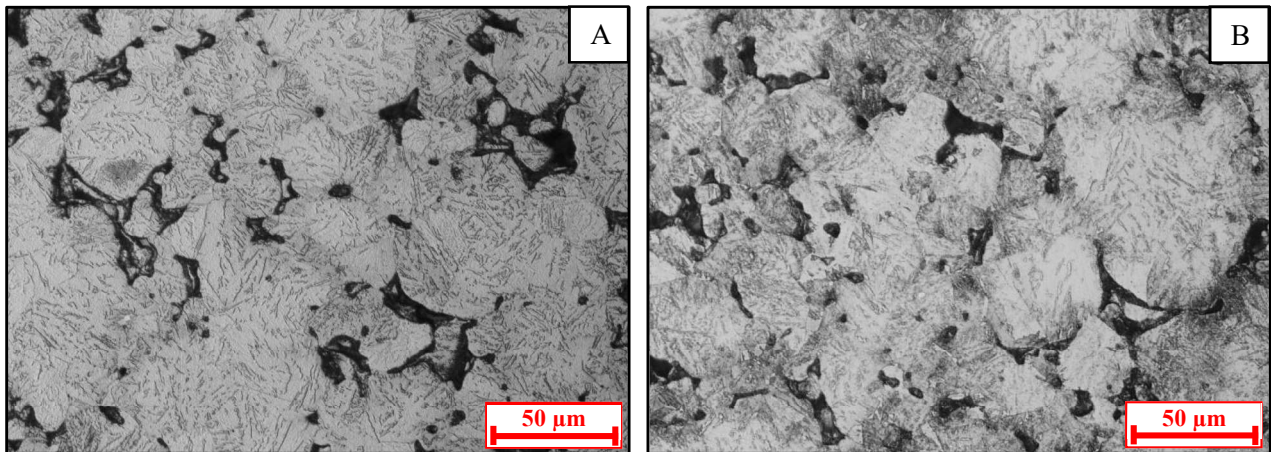


**Figure 7: Microstructure of green specimens, as-polished (200x). (A) regular copper. (B) fine copper**

Following copper growth, equivalent high-temperature shrinkages are observed (around 0.10%) and the cooling profiles are practically identical in both cases. The total high-temperature shrinkage of samples containing copper is less than the copper-free reference sample. This can be explained by the fact that, at the beginning of the plateau, shrinkage is probably still in competition with the growth caused by copper penetration.

The  $\gamma \rightarrow \alpha$  transformation seems to be modified by the presence of copper. Final microstructures are presented in Figure 8. Final dimensions observed are larger than green dimensions and confirm that copper caused a tangible growth.

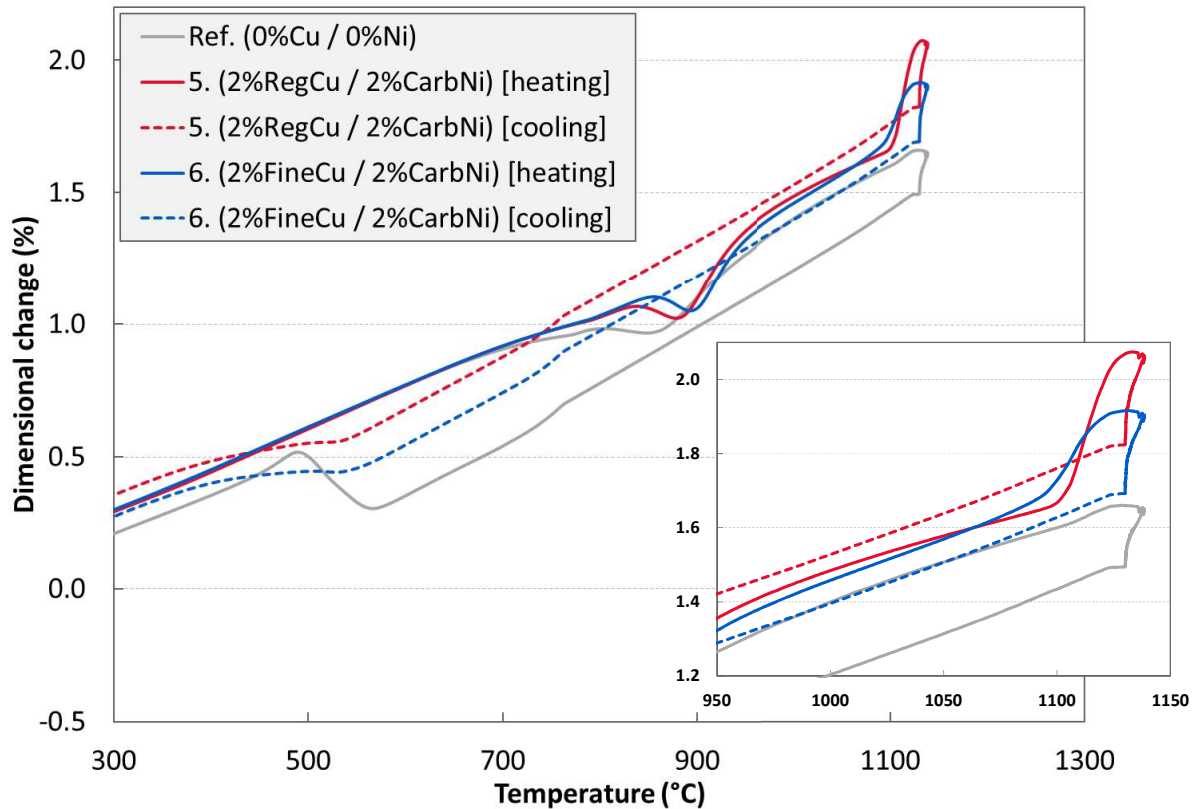
In the current case, the particle size distribution of copper seems to have a significant influence on the extent of the growth caused by copper penetration, fine copper promoting more growth.



**Figure 8: Microstructures of sintered specimens, etched with Nital (500x): (A) 2% regular copper / 0%Ni. (B) 2% fine copper / 0%Ni.**

#### Interaction between nickel and different copper additives

For this part of the study, samples with 2% copper and 2% nickel were prepared. Since the type of nickel (carbonyl vs reduced) appears to have no impact on the sintering behaviour, the dilatometry profiles shown in Figure 9 relate to samples containing only carbonyl nickel with the different types of copper.



**Figure 9 : Dilatometry curves of samples containing 2% of different types of copper and 2% of carbonyl nickel (FLNC-4405)**

Up to the melting temperature of copper, the expansion behaviour is similar to previous profiles shown in this study and earlier comments still apply here. However, the growth associated with copper adopts a singular shape in FLNC-4405 formulations when comparing with samples containing only copper or only nickel.

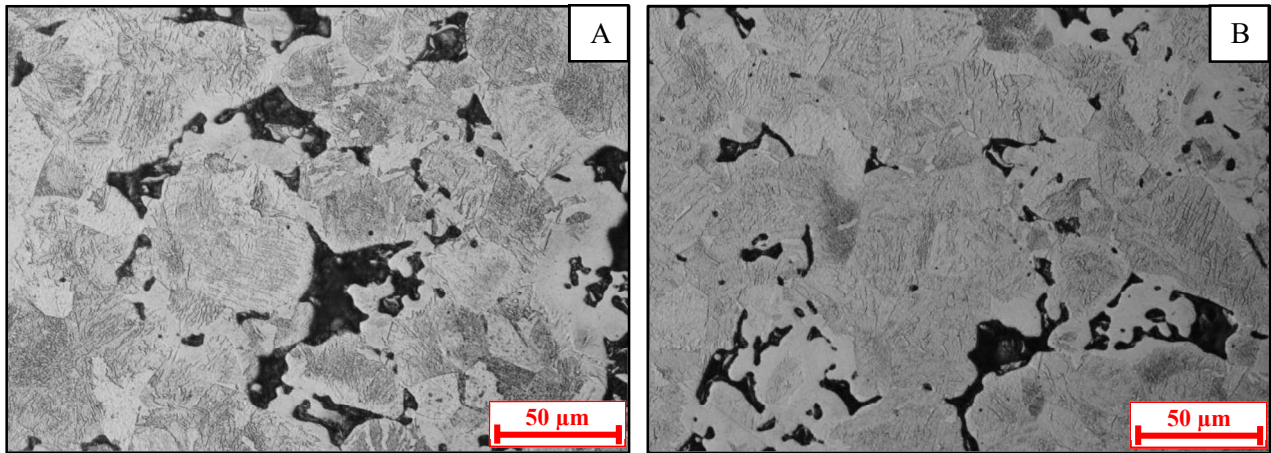
In the case of fine copper, clear differences are noticeable when comparing with the nickel-free sample (Figure 6). First, the beginning of the growth caused by copper appears at a higher temperature ( $\sim 1093^{\circ}\text{C}$  vs  $\sim 1083^{\circ}\text{C}$ ). Second, the total amplitude of this growth (0.23%) is significantly lower than what was observed with the nickel-free specimen (0.37%). The rate of this growth is similar with and without the presence of nickel ( $\sim 0.007\%/^{\circ}\text{C}$ ).

A very different kinetic is observed for regular copper. Strikingly, in the presence of nickel, the total growth is larger (0.42%) than the nickel-free specimen (0.32%) and the rate of growth is approximately 50% more pronounced ( $0.011\%/^{\circ}\text{C}$  vs  $0.007\%/^{\circ}\text{C}$ ). The temperature where swelling appears is similar with and without nickel ( $1095\text{-}1096^{\circ}\text{C}$ ).

Moreover, it is important to mention that the end of the growth regime appears to flatten at around  $1130^{\circ}\text{C}$ , just before reaching the sintering temperature, in both cases whereas in the case of nickel-free formulation the growth was still noticeable when reaching the maximum temperature ( $1138^{\circ}\text{C}$ ). It was postulated that the swelling effect of copper is probably still active during the early stages of the fixed

temperature plateau, competing with high-temperature shrinkage. In the current case, the presence of nickel seems to limit the growth as temperature reaches the maximum value.

When the sintering temperature is reached, shrinkage occurs: 0.22% in the case of fine copper and 0.25% in the case of regular copper, which is slightly lower than samples containing only 2% nickel (0.28%). The rest of the cooling response is similar in both cases and the  $\gamma \rightarrow \alpha$  transformation is, as expected, significantly affected by the presence of alloying elements when compared to the reference sample. Final microstructures are presented in Figure 10 and again show the presence of some residual austenite areas. The pearlite also seems to be finer than with the specimens containing only copper, only nickel or none of the two.



**Figure 10: Microstructures of sintered specimens, etched with Nital (500x): (A) 2% regular copper / 2% carbonyl nickel. (B) 2% fine copper / 2% carbonyl nickel.**

A possible explanation for the observed behaviour at high temperature is postulated in the following lines. First, the reader shall recall that nickel and copper have complete solubility. Pure copper melts at around 1083°C. As soon as copper melts, nearby nickel particles interact with the liquid phase and quickly go into solution. The melting temperature of a copper-nickel liquid phase increases with the amount of nickel. As an example, a liquid phase containing 5% nickel starts to melt at around 1105°C. At 1130°C, the copper-nickel alloy is fully solid when the concentration of nickel exceeds 12%. Also, it has been stated before that a copper-nickel liquid has a lower wetting angle than pure liquid copper [5]. These remarks are important when analyzing the sintering kinetics of sample containing both copper and nickel.

In the case of fine copper, once the temperature exceeds 1083°C, small liquid copper pools appear throughout the compact and quickly start to spread and to react with nearby nickel particles. Since these pools are small, the concentration of nickel in the liquid quickly increases. As the melting temperature of the liquid phase rises with increasing concentration of nickel, the spreading of the liquid is stopped. Actually, there is a competition between impeding the liquid spreading as the melting temperature increases and the better wettability caused by the presence of nickel in the liquid. All of that happens while the temperature of the furnace rises. Also, as fine copper and carbonyl nickel have approximately the same particle size, the spatial distribution of these additives should be very similar. In that sense, the concentration of nickel in the spreading liquid phase must be significant. Then, it can be postulated that the liquid phase travels in an intermittent manner into pore channels as it encounters nickel particles, going into solution and momentarily freezing the liquid phase. It would then explain why the growth

associated with grain boundary penetration and volume diffusion is delayed in time in the presence of nickel and why the amplitude of this growth is lower than in the nickel-free case. The following shrinkage observed at high temperature is still driven by the total surface energy of the compact. In this case, since significant amounts of nickel went into solution in the liquid phase, there is less fine particles contributing to this driving force. Consequently, the extent of shrinkage (0.22%) is smaller than the specimen containing only nickel (0.28%).

The use of regular copper with larger particles ( $d_{50} \sim 50\text{-}60 \mu\text{m}$ ) creates larger liquid copper pools, which are located further apart in the compact. Following the melting of copper, liquid starts to spread and to encounter nickel particles. But in this case, the amount of nickel going into liquid solution is small compared to the total local volume of melted copper, so the melting temperature of this liquid slowly rises and never catches up with the continuously rising temperature of the furnace: liquid phase stays liquid. The small presence of nickel in the liquid phase could also contribute to a better spreading since it should increase the wettability of the liquid. This could explain the faster rate of growth observed here. Finally, in this case, high-temperature shrinkage (0.25%) is only slightly lower than the sample containing only nickel (0.28%) but higher than the sample containing nickel and fine copper (0.22%). Again, these differences can be explained by the fact that only small amounts of nickel particles went into solution in the liquid phase.

As the mechanisms proposed here involve the spreading of a liquid phase, it is expected that the heating rate has a strong impact on the spreading kinetics of the liquid phase, and to a further extent, on the dimensional behaviour during the course of sintering. Future work could involve the use of different profiles and microstructural characterization of samples produced during interrupted dilatometry trials to observe and describe the movement of the different species in the compact. Special attention should be given to the range of temperature between 1080°C and 1120-1130°C where the effect of copper on dimensional change is dominant and where there is a more active interaction between nickel and copper.

## **CONCLUSIONS**

A dilatometry study was performed to evaluate and quantify the effect of particle size of copper and the morphology of nickel particles on the in-situ dimensional change of PM steels. Singular expansion behaviours were observed in the presence of both copper and nickel additives. Possible interactions mechanisms were suggested to explain the dimensional change. The following conclusions can be drawn from this study:

1. The morphology of nickel particles does not seem to have a significant impact on the dimensional change during the specific sintering cycle investigated (without copper).
2. It was observed that the use of fine copper leads to larger swelling at high temperature and that the growth related to copper begins earlier than with a coarser copper.
3. Singular expansion behaviours were observed for samples containing both copper and nickel, suggesting that an interaction between copper and nickel exists, especially in the range of temperature where copper is in the liquid phase.
4. It was suggested that the interaction between fine copper and nickel impeded the spreading of the liquid phase in the compact causing limited swelling.

5. On the other hand, the interaction between coarse copper and nickel seemed to enhance the liquid phase spreading leading to larger swelling.

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