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The Dimensional Change in the Sintering of a Plain Iron Powder and High-Alloyed Iron Powder Mixtures

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Abstract: In plain iron powder or powder mixtures sintering process, it is very important to control the dimensional change. In the sintering associated events, such as lubricant removal, atomic motion and phase transformation could change dimensions over a wide range. Dilatometric analysis has shown that most contributions in the dimensional change in the sintering of iron powder mixtures were due to the combination of several effects occurring in all stages of the processing, including the heating and cooling stages. The present paper has the objective of studying the dimensional behavior and to determine the transformation temperature of a composite powder mixture of a plain iron powder with various additions of high-alloyed steel, carbon, nickel and lubricant powders, during sintering by dilatometric analysis.

Introduction

Many alloying techniques have been designed with the purpose of enhancing the microstructural and mechanical properties of sintered steels. Among those, the mixing of high compressible iron powders with certain amounts of high-alloyed iron powders plus carbon and nickel has been utilized [1]. In several applications, like in automotive industries, the increasing demand of this technique depends on a satisfactory performance, with the main advantage of being economical and producing high strength and precision parts. Thus, changes in the dimensions of compacts are an important concern in the sintering process.

The dilatometry is a technique for following dimensional changes during plain iron powder or powder mixtures powder sintering [2-6]. The dilatometric analysis also is used in the study of the iron powder mixtures phase transformation [2, 7]. Dimensional change occurs due to the change in the lattice structure, which is accompanied by shrinkage or expansion of volume. The lattice diffusivity in face-centered cubic Fe is lower than that in body-centered cubic Fe. The reasons for a slower sintering response in the austenite region are slower inherent lattice and grain boundary diffusion, due to more rapid grain growth in the austenite phase [7-10].

In the solid state sintering theories propose the particle free surface reduction is obtained, through temperature activated atom transport mechanisms. This leads to interparticle neck growth and results in the mechanical strengthening of the compacted powder. The system may exhibit densification or not during this process depending on the mechanism involved [11].

The vapor transport and surface diffusion mechanisms aid interparticle bonding and pore rounding but do not cause densification. Therefore, these mechanisms are not detectable by dilatometry. The volume and grain boundary mechanisms also obtain pore elimination and cause densification. Thus, these mechanisms are detectable by dilatometry [12].

The presence of a liquid phase during sintering may be a means of accelerating particle bonding in ferrous alloys. Some phase liquids cause swelling, while others stimulate the densification. The liquid-phase sintering is less dependent on atomic motion for iron, once diffusion rates are high through the liquid phase [13].

The present paper has the objective of studying the dimensional behavior of a composite powder mixture in a plain iron powder with various additions of high-alloyed steel, carbon, nickel and lubricant powders, during sintering by dilatometric analysis.

Materials and methods

An iron powder manufactured by Hoganas (designated as ABC 100.30) was mixed with a high-alloyed iron powder manufactured by Hoganas (designated as Cold PMo). The high-alloyed iron powder (Cold PMo) is compound by water atomized iron powder, Fe-10Mo prealloyed with 10% Mo (weight), carbon and iron phosphorus. Carbon addition was used in order to maintain the final composition in 0.4 and 0.7%, in weight. Nickel was used in the preparation of the mixtures in the compositions of the 2 and 5% (weight). The chemical composition, medium diameter (d) and apparent density (ad) of these starting powders are given in Table 1.

Table 1: Characteristics of the starting powders.

Powder type	**%C**	**%P**	**%Mo**	**%Ni**	**H_2 loss**	**%O_2**	**%Fe**	**d[µm]**	**ad[g/cm^3]**
Fe (plain iron))	0.004	-	-	-	0.040	-	Bal.	90.0	3.01
Fe (high alloyed)	1.10	0.55	9.75	-	0.25	-	Bal.	35.0	3.20
Ní	0.07	-	-	99.80	-	0.12	<0.01	4.6	2.07

Powder mixtures were based on the starting powders. The mixtures were processed for 1,800 seconds in a turbo-type mixer. The mixture compositions investigated are given in Table 2. An addition of 0.4% Kenolube was used as lubricant only in mixtures 1 – 8.

The starting powders and powder mixtures were single-axially cold-compacted under pressures of 600.10 ^{6} Pa. A thermal dilatometer (Netzch – 402S) was employed to compare the sintering curve of these mixtures and starting powders. The heating and cooling rates used were 10^{o}C/minute, under argon. The microstructure was observed by scanning electron microscopy (XL30, Philips).

Table 2: Chemical compositions (%weight) of the mixtures.

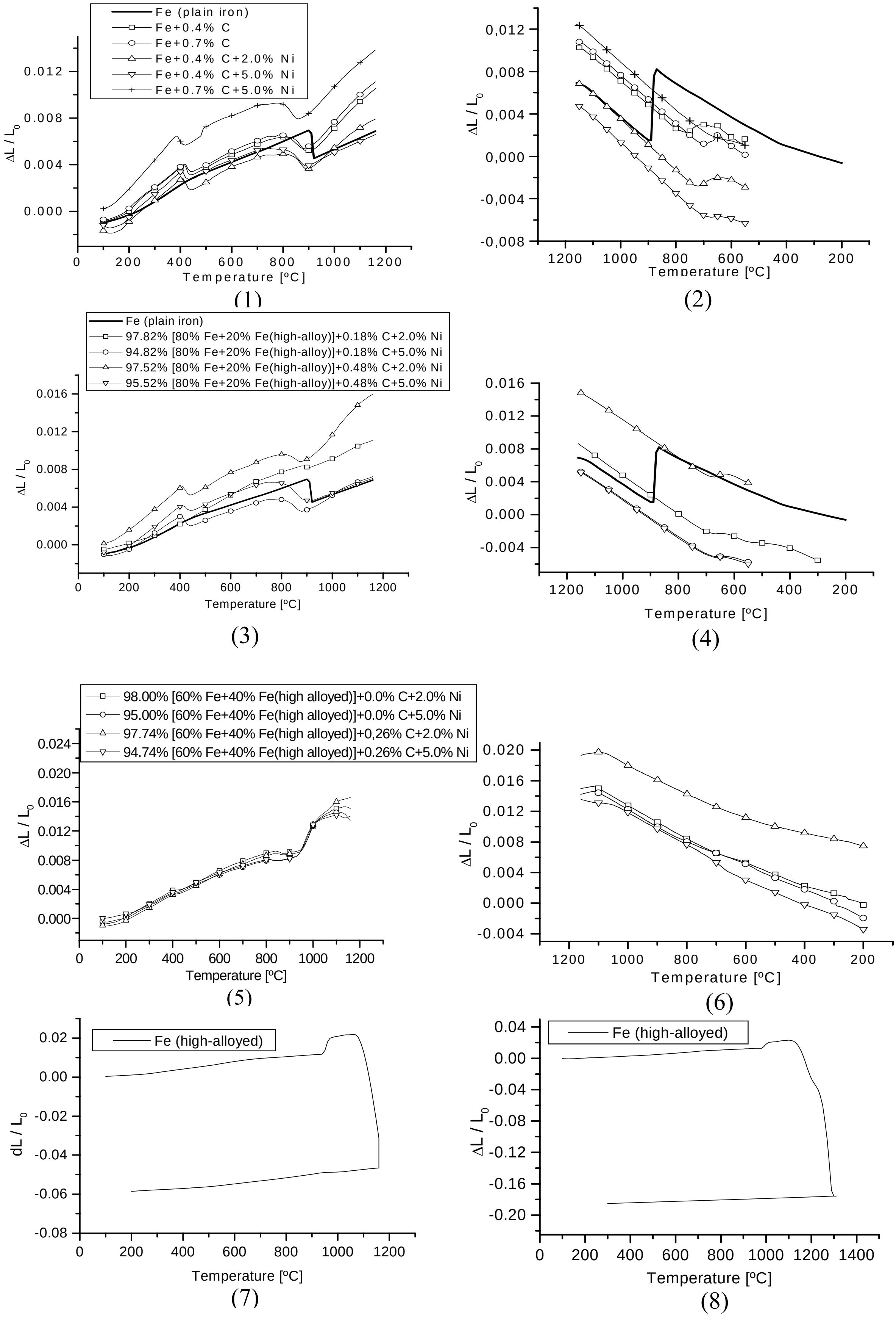
Mixture	Chemical compositions
1	97.2% Fe + 2.0% Ni + 0.4% C
2	94.2% Fe + 5.0% Ni + 0.4% C
3	96.9% Fe + 2.0% Ni + 0.7% C
4	93.9% Fe + 5.0% Ni + 0.7C% C
5	97.82%[80%Fe + 20%Fe(high alloyed)] + 2.0%Ni + 0.18%C
6	94.82%[80%Fe + 20%Fe(high alloyed)] + 5.0%Ni + 0.18%C
7	97.52%[80%Fe + 20%Fe(high alloyed)] + 2.0%Ni + 0.48%C
8	94.52%[80%Fe + 20%Fe(high alloyed)] + 5.0%Ni + 0.48%C
9	98.0%[60%Fe + 40%Fe(high alloyed)] + 2.0%Ni + 0%C
10	95.0%[60%Fe + 40%Fe(high alloyed)] + 5.0%Ni + 0%C
11	97.74%[60%Fe + 40%Fe(high alloyed)] + 2.0%Ni + 0.26%C
12	94.74%[60%Fe + 40%Fe(high alloyed)] + 5.0%Ni + 0.26%C

Results and discussion

Figures 1 to 8 depict the measured variation curves represented as $\Delta L/L_0$ versus temperature, for the twelve mixture compositions given in table 2 plus two starting iron powders (plain iron and high-alloyed iron). In figures 1, 3 and 5 the heating curves obtained are showed, while in figures 2, 4 and 6 the cooling curves are indicated. In figures 7 and 8, the dimensional variations obtained from the sintering cycle are presented, including the isothermal level in high-alloyed iron powder. In the Fig. 1 and 3 a first inflection of about 400ºC is showed, due to the lubricant evaporation. Heat melts the lubricant and the evaporation starts at 400ºC, and almost all the lubricant is gone by 500ºC. The lubricant removal causes reduction in the internal pressure and volume.

A second inflection occurs at temperatures near 900ºC owing to α-ferrite to γ-austenite transformation. A contraction undergone by a plain iron powder is showed in the dilatometric curve of Fig.1 and 3. The temperature interval at which the ferrite plus perlite-to-austenite transformation starts and ends was 900 to 920ºC. The curves of mixtures 1 to 4 showed an increase in the transformation temperature interval of 770 to 900ºC. The phase transformation temperature decreased after nickel alloying and carbon addition. The carbon and nickel are stabilizers of the austenite phase. Moreover, the phase transformation did not take place at a fixed temperature because the microstructure was heterogeneous.

The phase transformations temperature interval α to γ were higher for the compositions, with an amount of 20 and 40% (high-alloyed iron powder), as observed in figures 3 and 5. The largest temperature intervals in the mixtures are a consequence of microstructural heterogeneity. Regions with larger amounts of γ-stabilizers (carbon and nickel) cause the transformation temperature reduction. Areas with a greater amount of α-stabilizers (phosphorus and molybdenum) increase the ferrite stability field, increasing the transformation temperature.



Figures 1 to 8: The measured dimensional variation curves ($\Delta L / L_0$) X Temperature [ºC] for all mixtures of Table 2 plus starting powders.

In the cooling curves of figures 2, 4 and 6 transformation temperatures γ to α for plain iron distinct from those for the mixtures are observed. The expansion in plain iron transformation occurred in a temperature practically constant, around 890^{o} C. The transformations in mixtures 1 to 12 took place in larger temperature intervals and in lower temperatures. In the processing conditions used, the presence of γ-stabilizers was preponderant to the α-stabilizers for reducing the transformation temperature.

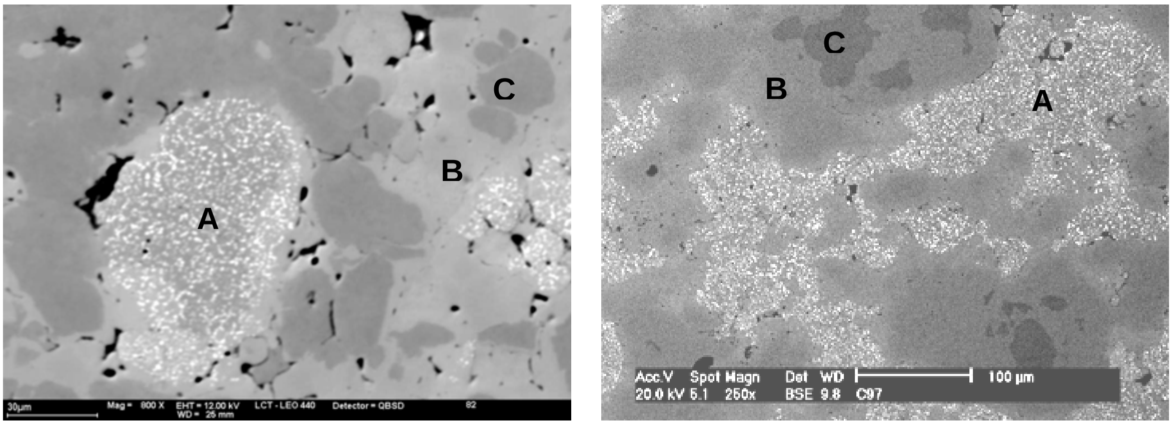
The nickel additions provoked higher contractions values in the plain iron and higher swelling in the high-alloyed mixtures. The compositions with 0.7% (weight) presented a tendency of swelling in comparison with the 0.4%C mixtures

The dimension variations of the sintering, independently from the mixture, were below 1%. Previous studies have shown that surface diffusion is the dominant sintering mechanism for iron but it does not contribute to densification [11]. Despite the regions with the presence of transient liquid phase, as the Fe_3P phase, the amount of liquid phase formed was not sufficient to generate significant dimensional variations. Under these sintering conditions, the liquid phase is not present in enough amount and for enough time to greatly influence densification. In the isothermal conditions of the mixtures sintering, a significant contraction in relation to time was not detected.

The high-alloyed iron powder was exception, as seen in figures 7 and 8, which show contractions of 6 and 20 % for the temperatures of 1150 and 1300^{o} C, respectively. The high-alloyed iron powder contractions occurred, mostly, in the isothermal level of sintering. In the Fe-P system, there is a low-melting eutetic phase at about 1050º C which has a high solubility for iron. The liquid phase in the iron-phosphorus is only a transient liquid phase but had a strong effect to enhance iron powder sintering. For certain phosphorus concentrations, the α-iron is stabilized at the sintering temperature. The presence of α-iron allows rapid self-diffusion during the sintering.

Transient liquid phase sintering (TLPS) occurs when the liquid phase, such as Fe_3P in a mixture of iron powder, is absorbed by the solid phase during sintering. The TLPS process depends on sintering conditions such as the amount of liquid formed, and the time it lasts. The liquid flows between the powder particles, filling pores and causing densification by capillary action and through the provision of a fast diffusion pathway. Shrinkage occurs by particle rearrangement when the liquid volume is high enough and the green density is low enough, by the particles changing shape to allow better packing, by pore filling and by solid state sintering, if a solid skeleton forms. The compacted contraction suggests volume diffusion and via grain boundary, as a sintering mechanism.

Scanning electron micrographs are shown in Figures 9 and 10. The regions were identified using back scattered electrons. The sintered specimens had heterogeneous microstructures. A heterogeneous distribution of the alloy elements (molybdenum and nickel), during sintering, was observed and identified in figures 9 and 10 in areas "A", "B", and "C". The microstructures consisted of a phases mixture such as: pearlite, bainite, martensite, retained austenite and pores. The microstructural heterogeneity contributes to larger temperature intervals for the occurrence of the complete phase transformation, during heating and cooling and also for smaller dimensional variations, observed in the curves obtained by dilatometry.



(A) (B)

Figures 9 and 10: SEM-BSE (back scattering), (A) mixture12 and (B) mixture 20; “A”, region Fe-Mo; “B”, rich Ni region and “C”, plain iron powder area.

Conclusions

1. The addition of carbon and nickel (γ-stabilizers) in iron powder decreased the α → γ and γ→α phase transformation temperature.
2. The addition of high-alloyed iron powder to plain iron increased the α → γ phase transformation temperature and decreased γ → α phase transformation temperature
3. The presence of certain phosphorus concentrations in the high-alloyed iron powder allows both the stabilization of α-iron and the rapid self-diffusion during the sintering.
4. The microstructure was heterogeneous mainly because of the heterogeneous distribution of the alloy elements (molybdenum and nickel)

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