Fabrication of metallic iron green pieces by colloidal processing techniques in aqueous media

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Introduction:

- The development of Fe-based composite materials is motivated by the need of a replacement of Cobalt and Nickel in metal ceramics, due to their toxicity.
- In this work we have studied the colloidal behavior of Fe in aqueous suspensions. This study is focused on the obtaining of suspensions able of being processed with Ti(C,N) high ceramic reinforcement (50%).
- In contrast to the difficulty of micrometric powders processing by pressing, the study of colloidal dispersion from homogeneous suspensions arises as a feasible alternative.
- Aqueous colloidal processing of these materials allows obtaining homogeneous composite materials with complex shapes and structures. The rheological study of suspensions allows to establish the necessary flow conditions during processing

Powder Characterization:

Characterization of the Fe powder together with Ti(C,N) is summarized in Table 1

	Element Analysis [wt%]			Distribution Size [µm]		Specific surface area [m²/g]	Theoretical Density [g/cm³]
	C	N	O	[D_v50]	d_{BET}		
Fe	<0.1	<0.01	<0.5	3.47 ± 0.01	1.2 ± 0.1	0.05 ± 0.01	4.4 ± 0.1
Ti(C,N)	10.5	10.5	<1.0	3.52 ± 0.01	0.4 ± 0.1	1.70 ± 0.01	5.1 ± 0.1

Table I. Characteristics of Fe and Ti(C,N) powders.

Fe powder presents a spherical morphology and a narrow grain distribution, Ti(C,N) powders show an irregular shape (Figure 1).

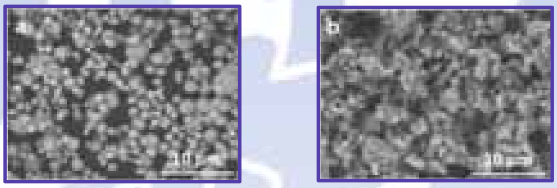


Figure 1 SEM micrographs of a) Fe and b) TiC,N)

Suspension Characterization:

Colloidal stability of Fe in aqueous media was determinate by zeta potential measurements as a function of pH as shown in Figure 2. For pH > 9 superficial charge allows to prepare stable suspensions with high solid content which are stable due to electrostatic repulsive forces.

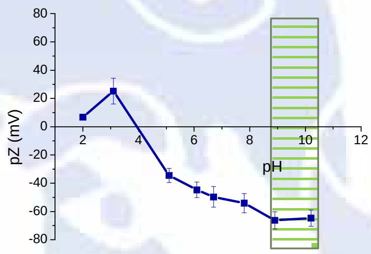


Figure 2. Zeta potential of Fe

Corrosion:

Suspensions 10g/l of Fe in water at different pH values were prepared, and filtered after 15 days. The wastewaters are shown in figure 3. According to the different colors depending on the amount of Fe^{3+} dissolve, the lowest concentration of Fe^{3+} is observed at pH 11.

Figure 3. Oxidation of Fe vs. pH

Rheology:

According to zeta potential measurements and non-oxidation conditions, all slurries where formulated at pH 11 with 45% vol. solid contents

- A milling study of the suspensions of Fe was conducted to determine the optimal time to obtain homogeneous (Figure 4).
- In order to optimize the rheological behavior of Fe slurries, addition of citric acid and Nitrilotriacetic acid (NTA) has been studied (Figure 5).

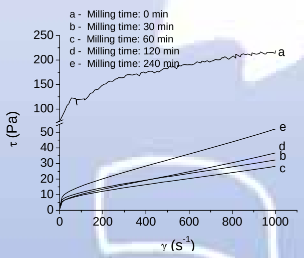


Figure 4 Flows curves Fe 45% vs. milling time Mill : nylon balls of 15 mm in diameter.

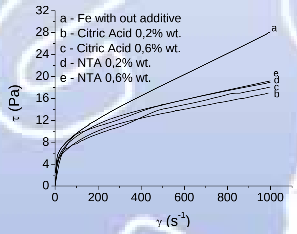
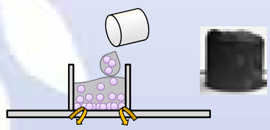


Figure 5. Flows curves Fe 45% with citric acid and NTA

Iron matrix metal ceramic:

- For this purpose the combined dispersion of Fe and Ti(C,N) was carried out with 0.2%wt citric acid. Figure 6 shows the flow curves under optimized conditions
- A green body was processed by slip casting . Once dried, the sample was characterized by Hg picnometry and SEM (Figure 7).



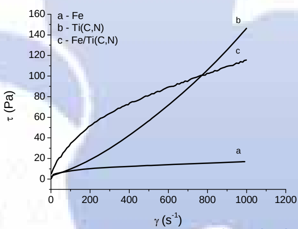


Figure 6. CR flows curves of a) Fe b) Ti(C,N) and c) Fe-Ti(C,N) 45%volume slurries with citric acid (0,2%wt).

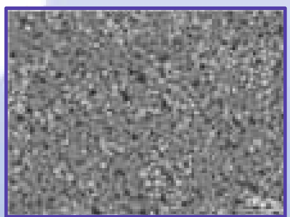


Figure 7. Fe/Ti(C,N) SEM micrograph of unpolished surface

Conclusions

- Suspensions of Fe micrometer in water were optimized. Maximum dispersion by electrostatic repulsion mechanism and Fe corrosion inhibition were archived at pH 10-11.
- Through rheogical studies, have been formulated stables high content slurries of Fe in water (45%vol). Milling and citric acid addition (0,2% wt) improve fluidity of the suspension.
- Fe aqueous suspensions allow the dispersion together with Ti(C,N)suspensions, which presents a rheological behavior that can be processed by slip casting (region from 125 to 100 s^{-1}). Green bodies thus obtained have a density of 3.14 g/cm³ (49% relative to theoretical density) and a high degree of dispersion of the ceramic phase in the metal matrix.

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