

Structural study of Fe/SiO₂, Fe-Ni/SiO₂ and Fe-Co/SiO₂ nanocomposites

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Abstract

The present work examines Fe, Fe-Ni and Fe-Co nanocomposites supported on silica, prepared by impregnation, followed by calcination and thermal H₂- treatment. Several techniques, such as SEM and XRD are used for the characterization of the samples at different steps of their elaboration. After impregnation, the X-rays diffraction reveals the formation of iron, nickel and cobalt silicide (FeSi₂, FeNiSi, FeCoSi). After calcination, the XRD pattern present the features of iron, nickel and cobalt oxides (Fe₂O₃, Fe₂NiO₄, FeCoO₄) with an average size of 10-15nm.

Key words: nanocomposites, structural properties, Fe, Ni, Co, SiO₂, FeSi₂, Fe₂CoSi, FeNiSi, Fe₂NiO₄, Fe₂CoO₄, Metal/Support interaction.

Introduction

The nanomaterials are covering a broad range of topics in applied science and technology because of their original structural, electrical, magnetic and optical properties, different from that of bulk state. Particularly, there has been an increased interest in nanoscaled iron and its nanoalloys due to their several potential applications, such as magnetic resonance imaging for medical diagnosis [1] (Fe), controlled drug delivery [2] (Fe-Co) and catalysis [3] (Fe-Ni).

Experimental

The solvent was water of ultra-high purity. The chemicals were supplied with highest purity available and used as received: from Merck for nickel formate (NiH₂O (COOH)), Biochem for iron sulfate (FeSO₄7H₂O) and cobalt nitrate (Co (NO₃)₂6H₂O). The silica support was from Degussa.

In a first step, the conditions of the adsorption of the metal precursor on silica are optimized. The iron, nickel and cobalt ions are fixed on SiO₂ surface by ionic exchange in wet conditions. In a second step, the samples are calcined at various temperatures (T = 200-700°C) during 1h30 min. After calcination, the samples are reduced under H₂ at 350 or 500°C.

Results and discussion

After impregnation, the XRD patterns present, in addition to broad peak relative to amorphous SiO₂, reactive metal/support phases: FeSi₂,

FeNiSi, FeCoSi, respectively for Fe/SiO₂, Fe-Ni/SiO₂ and Fe-Co/SiO₂. All of these phases have orthorhombic structure. After calcinations for Fe/SiO₂, the XRD study reveals the formation of nanosized of α -Fe₂O₃ phase at 500°C. For Fe₅₀Ni₅₀/SiO₂ calcined at 700°C, the XRD pattern presents the features of FeNi₂O₄ oxide phase. (15 nm sized). The cell parameters are reported on table 1. In the case of Fe₅₀Co₅₀/SiO₂, the XRD study reveals the presence of bimetallic oxide phase Fe₂CoO₄ (17 nm sized) at T \geq 500°C.

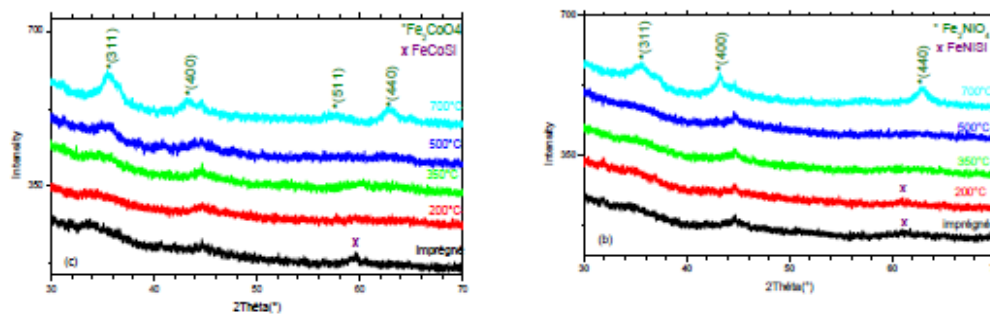


Figure 1: XRD patterns of: (a) Fe/SiO₂, (b) Fe-Ni/SiO₂ and (c) Fe-Co/SiO₂.

Table 1. Structural parameters and particle size of Fe/SiO₂, Fe-Ni/SiO₂ and Fe-Co/SiO₂

The samples		T (°C)	Phases	Cell parameters (nm)	Structure	D (nm)
Fe/SiO ₂	After impregnation	80	FeSi ₂	a = 0,4 b = 7,81 c = 7,52	Orthorhombic	10
	After calcination	700	Fe ₂ O ₃	a = 4,36	Rhombohedral	15
Fe-Ni/SiO ₂	After impregnation	80	FeNiSi	a = 5,00 b = 3,75 c = 7,67	Orthorhombic	11
	After calcination	700	Fe ₂ NiO ₄	a = 8,27	CC	14
Fe-Co/SiO ₂	After impregnation	80	FeCoSi	a = 4,89 b = 3,65 c = 7,15	Orthorhombic	13
	After calcination	700	Fe ₂ CoO ₄	a = 8,20	FCC	17

Conclusion

The structural study of silica-iron based nanocomposites reveals the formation of metal/support interaction phases: iron silicates: FeSi₂, FeNiSi and FeCoSi, after ionic exchange and iron oxides: Fe₂O₃, Fe₂NiO₄ and Fe₂CoO₄, after calcination. Their size is about 10-17 nm. Due to the properties of these phases, the studied composites have a choice place for several applications in magnetism and catalysis.

References

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