Structural study of Fe/SiO_2, $Fe-Ni/SiO_2$ and $Fe-Co/SiO_2$ 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 H_2- 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 ($FeSi_2$, FeNiSi, FeCoSi). After calcination, the XRD pattern present the features of iron, nickel and cobalt oxides (Fe_2O_3, Fe_2NiO_4, $FeCoO_4$) with an average size of 10-15nm.

Key words: nanocomposites, structural properties, Fe, Ni, Co, SiO_2, $FeSi_2$, Fe_2CoSi, FeNiSi, Fe_2NiO_4, Fe_2CoO_4, 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

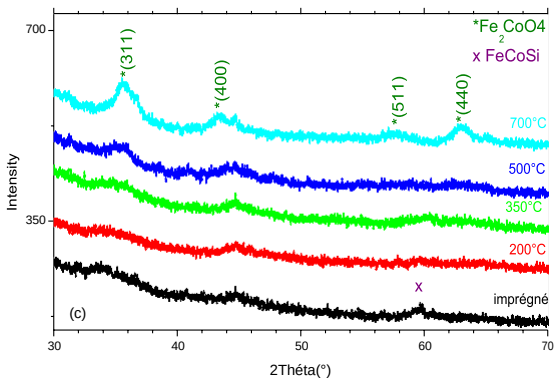
The solvent was water of ultra-high purity. The chemicals were supplied with highest purity available and used as received: from Merck for nickel formate (NiH_2O (COOH)), Biochem for iron sulfate ($FeSO_47H_2O$) and cobalt nitrate (Co $(NO_3)_26H_2O$). The silica support was from Degussa.

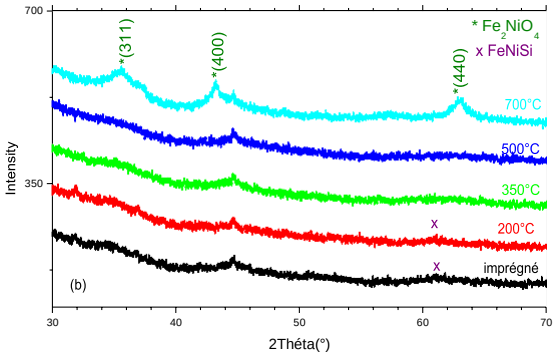
In a first step, the conditions of the adsorption of the metal precursor on silica are optimized. The iron, nickel and cobalt ions are fixed on SiO_2 surface by ionic exchange in wet conditions. In a second step, the samples are calcined at various temperatures (T = 200-700°C) during 1h30 min. After calcination, the samples are reduced under H_2 at 350 or 500°C.

Results and discussion

After impregnation, the XRD patterns present, in addition to broad peak relative to amorphous SiO_2, reactive metal/support phases: $FeSi_2$, FeNiSi, FeCoSi, respectively for Fe/SiO_2, $Fe-Ni/SiO_2$and $Fe-Co/SiO_2$. All of these phases have orthorhombic structure

After calcinations for Fe/SiO_2, the XRD study reveals the formation of nanosized of α-Fe_2O_3 phase at 500°C. For $Fe_{50}Ni_{50}/SiO_2$calcined at 700°C, the XRD pattern presents the features of $FeNi_2O_4$ oxide phase. (15 nm sized). The cell parameters are reported on table 1. In the case of $Fe_{50}Co_{50}/SiO_2$, the XRD study reveals the presence of bimetallic oxide phaseFe_2CoO_4 (17 nm sized) at $T \geq 500$°C.


*Fe2CoO4
x FeCoSi
*(311)
*(400)
*(511)
*(440)
700°C
500°C
350°C
200°C
x
imprégné
(c)
Intensity
700
350
30
40
50
60
70
2Théta(°)


* Fe2NiO4
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*(311)
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700°C
500°C
350°C
x
200°C
x
imprégné
(b)
Intensity
700
350
30
40
50
60
70
2Théta(°)

Figure 1: XRD patterns of: (a) Fe/SiO_2, (b) $Fe\text{-}Ni/SiO_2$ and (c) $Fe\text{-}Co/SiO_2$.

Table 1.**S**tructural parameters and particle size of Fe/SiO_2, $Fe\text{-}Ni/SiO_2$ and $Fe\text{-}Co/SiO_2$

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_2O_3	a = 4,36	Rhombohedric	15
$Fe\text{-}Ni/SiO_2$	After impregnation	80	FeNiSi	a = 5,00 b = 3,75 c = 7,67	Orthorhombic	11
	After calcination	700	Fe_2NiO_4	a = 8,27	CC	14
$Fe\text{-}Co/SiO_2$	After impregnation	80	FeCoSi	a = 4,89 b = 3,65 c = 7,15	Orthorhombic	13
	After calcination	700	Fe_2CoO_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: $FeSi_2$, FeNiSi and FeCoSi, after ionic exchange and iron oxides: Fe2O3, Fe_2NiO_4 and Fe_2CoO_4, 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

[1] Ross, C.Annu.Rev.Mater.Res.2001, 31,203.

[2] Bin Kang, Shu-quan Chang, Yao-dong Dai, Da Chen. Radiation physics and chemistry 76 (2007), 968-973. [3] Wenhua Wang, Hey Wang, Yong Yang, Shubian Jiang.International journal of hydrogen energy 37-2012), 9058-9066.