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Novel synthesis and applications of yttrium silicates from a silicone resin containing oxide nano-particle fillers

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Abstract

In this paper, a novel method for the synthesis of yttrium silicates is presented. Such silicates are well known to be promising materials for protecting various substrates against high temperature oxidation, but they can typically be produced only after quite complicated processing. The use of preceramic polymers, in which a silicate article is obtained by direct thermal treatment in air of nano-composites consisting of silicone resins containing suitable oxide nano-particles, is a valid alternative, since the desired phases, i.e. Y-monosilicate ($Y_2O_3{\cdot}SiO_2$) and Y-disilicate ($Y_2O_3{\cdot}2SiO_2$) can be obtained by treatments at low temperature (1000–1400 °C). Y-disilicate could be employed for the manufacturing of dense and thick coatings on SiC foamed substrate, by simply dipping the substrates into silicone suspensions, before ceramic conversion. Y-monosilicate, that could be also useful for coatings, was found to exhibit promising characteristics, when doped with Eu_2O_3, making it of interest for application as red phosphor for LEDs.

Keywords: B. Nanocomposites; D. Silicate; E. Thermal applications; E. Functional applications

1. Introduction

Among oxide ceramics, silicates are well known to possess a high thermal and chemical stability, due to their characteristic partially covalent bonding and limited ionic interdiffusion. Such features, however, pose some difficulties in the overall processing, since they are related to a poor solid state sinterability [1,2]. Silicate ceramics with limited secondary glassy phases are generally obtained by spark plasma sintering [2–4] or chemical vapor deposition [5,6], i.e. by processes so complicated and expensive that extensive applications are greatly limited.

The present paper aims at providing a novel method for the synthesis of yttrium silicates based on the use of silicone resins containing oxide nano-particle fillers. Silicone/oxide nano-composites have been recently proven to be valid precursors for silicates of high technological relevance, such as mullite ($3Al_2O_3{\cdot}2SiO_2$), wollastonite ($CaO{\cdot}SiO_2$) and zircon ($ZrO_2{\cdot}SiO_2$) [7–12]. Silicones act as sources for silica, reacting easily with the nano-sized dispersed phase due to the quasi molecular mixing that can be achieved during the forming of the component. In fact, filler incorporation is greatly favored by the easy dissolution of silicones in common solvents, such as acetone or isopropyl alcohol, leading to solutions in which nano-particles can be put in suspension. Fine nano-composite powders, obtained by gentle grinding of the solid residue left upon drying of suspensions, can be employed for the final shaping, carried out by cold or warm pressing or even by extrusion [13]. The produced preceramic article, can be then directly transformed into a ceramic component by controlled treatment in air. Although some shrinkage occurs, a proper formulation of fillers allows the production of dense (especially in conjunction with the presence of some sintering aids) and uncracked samples [9,12], with no significant shape distortion. If compared to the sol–gel route, the PDCs route is characterized by a generally higher ceramic yield and a better control of the viscosity of the precursors, which in turn could give a much more limited global shrinkage of the materials during their processing and, above all, the possibility of applying conventional plastic shaping technologies for an easy manufacturing of ceramic components.

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Yttrium silicates are subjected to a the growing interest for materials suitable for high temperature applications [14]. Yttrium monosilicate, $Y_2O_3{\cdot}SiO_2$ (or Y_2SiO_5, Y_2O_3/SiO_2 molar ratio equal to 1, later called Y-MS), and yttrium disilicate, $Y_2O_3{\cdot}2SiO_2$ (or $Y_2Si_2O_7$, Y_2O_3/SiO_2 molar ratio equal to 0.5, later called Y-DS) are relatively new refractory silicates. Y-MS is practically monophasic, being subjected only to a dislocative (i.e. martensitic) transformation between the X1 phase (low temperature phase) and the X2 phase (high temperature phase) [15]. Y-DS, on the other hand, features many polymorphs (y, α, β, γ, δ, z) [16,17]; all the forms are stable in a certain temperature range, but high temperature polymorphs may be retained at room temperature, due to extremely slow phase transformations [15]. A particular polymorph may be associated to a specific processing procedure [17]: for instance, whereas sol–gel processing favors the formation of the α-phase [18,19], hydrothermal synthesis is known to promote the development of the y-phase [20].

For intermediate compositions (Y_2O_3/SiO_2 molar ratio between 0.5 and 1), mono- and disilicate may coexist in their relative polymorphs [15]. The interest for yttrium silicates is due to the high melting points and, above all, their relatively low coefficients of thermal expansion and Young's modulus values, which afford to these ceramics a good thermal shock resistance [14]. In the case of Y-DS, the coefficient of thermal expansion is very close to that of SiC ceramics, so that it has been widely recognized as a promising anti-oxidation coating phase [14,15,21]. Typically, the production of these coatings rely on quite complicated processing procedures, such as plasma spraying or CVD [14,15,21].

In this paper, we will firstly discuss the possibility of effectively producing yttrium silicates from silicone resins filled with nano-sized oxide particles. Then we will show the potential of this processing method for the manufacturing of anti-oxidation coatings on SiC substrates as well as the possibility of producing powders exhibiting a red luminescence under visible/near-UV radiation, when doped with Eu oxide, that could be used in LEDs.

2. Experimental procedure

A commercial polysilsesquioxane (Silres R MK, Wacker-Chemie GmbH, München, Germany) was dissolved in isopropyl alcohol and mixed with Y_2O_3 nano-sized powders (purity >99.95%, diameter 30–50 nm, Inframat Advanced Materials, Manchester, CT), in two Y_2O_3/silicone weight proportions (3.16 and 1.58) for the production of Y-MS and Y-DS, respectively. The solid content, in all dispersions, was kept constant, at about 20%. The weight ratios were obtained considering both the Y_2O_3/SiO_2 molar ratios in the silicates, being 1/1 or 1/2, and the ceramic yield of the preceramic polymer, intended as amount of amorphous silica left after heat treatment in air (84 wt%) [7,9,11]. All the dispersions were homogenized by magnetic stirring for 10 min, and then ultrasonicated for 20 min to reduce the size of the residual particles agglomerates, thus obtaining homogeneous and stable dispersions. After the evaporation of the solvent in an oven at 90 °C in air, the material obtained was first finely ground with mortar and pestle, to a size of about 100 μm, then put on alumina trays and subjected to thermal treatment in air, with target temperatures varying from 900 to 1500 °C, with holding time of 1 h and heating rate of 10 °C/min. The phase development was investigated by means of X-ray diffraction (Bruker AXS, D8 Advance, Karlsruhe, Germany) operating with CuKα radiation (0.15418 nm). The diffraction patterns were analyzed by means of the Match! program package (Crystal Impact GbR, Bonn, Germany), supported by data from the PDF-2 database (International Centre for Diffraction Data – ICDD, Newtown Square, PA). The density of the powders was measured by gas pycnometry (Micromeritics AccuPyc 1330, Norcross, GA).

SiC open-celled foams (10 ppi ERBISIC-R foams, Erbicol SA, CH) were used as substrates for coating experiments. The foams, produced by the replica method, are comprised of α-SiC powder embedded in a β-SiC/silicon matrix, and possess a bulk average density of 2.83 g/cm^3 about 86 vol% porosity, and a cell size of approximately 3 mm [22]. Coatings were applied by dipping the foams in an water-based emulsion, following a procedure previously applied to mullite coatings on SiC foams [23]. A silicone/Y_2O_3 suspension was prepared by using a low amount of solvent (2.2 ml for 1 g of solid), then cast drop-by-drop in distilled water, under vigorous magnetic stirring, followed by ultrasonication for 10 min. Before casting, water had been added with a non-ionic surfactant (Pluronic P65, BASF Corporation, Florham Park, NJ), in a water/surfactant weight ratio equal to 30; the relative amount between the suspension and the water was chosen in order to have a water/solid (silicone + filler) weight ratio equal to 3.

SiC foams were dipped and extracted several times in the emulsion: the deposited layer, after each coating operation, was stabilized at 300 °C in air for 5 min (by direct insertion in oven), while the emulsion was kept homogeneous by applying sonication. Any excess of coating medium, forming thick drops at the cell corners of SiC foams, was removed after dipping by spraying compressed air. The ceramization of the coating layer, i.e. the conversion of the polymer and the filler into Y-DS, was achieved by applying three thermal cycles in air, each following two depositions. Every cycle lasted 1 h at the maximum temperature, and the sample was heated with a 2 °C/min rate. The temperature reached during the first heating cycle was 600 °C, whereas the second and the third heating cycles reached 1400 °C. A white, compact deposit was produced after the heat treatments. Morphological characterization was performed by optical stereomicroscopy and scanning electron microscopy (FEI Quanta 200 ESEM, Eindhoven, NL).

The formulation suitable for yielding yttrium mono-silicate (Y-MS) was used for the preparation of luminescent powders. Y_2O_3 nanoparticles were partially replaced by Eu_2O_3 nanoparticles (purity >99.9%, diameter 45–58 nm, Cometox Srl, Milan, Italy), for an amount of 5 mol%, thus giving the final composition of $(Y_{0.95}Eu_{0.05})_2SiO_5 = Y_{1.9}Eu_{0.1}SiO_5$ instead of Y_2SiO_5. The overall preparation procedure was the same than that adopted for pure yttrium silicate, except for firing, which was conducted at 1000–1300 °C (heating rate of 10 °C/min), with a holding time of 1–3 h. Preliminary optical characterization was conducted by means of a spectrofluorometer (FP-6300, JASCO Ltd., Great Dunmow, UK), on powders ground and sieved to a maximum diameter of 2 μm, dispersed in distilled water.

3. Results and discussion

Fig. 1a reports the X-ray diffraction patterns of samples corresponding to a Y_2O_3/SiO_2 molar ratio equal to 1 (Y-MS formulation), illustrating the phase evolution with increasing processing temperature. At 900 °C the oxide filler remained substantially unreacted, Y_2O_3 (PDF#76-0151) being the only recognizable crystal phase; a slight temperature increase (1000 °C), however, led to the formation of Y-MS, in its low temperature form, X1 (PDF#21-1456). Increasing the firing temperature from 1200 °C, at which the formation of X1 phase was confirmed (since the patterns at 1000 °C, 1100 °C and 1200 °C are practically identical, the pattern at 1100 °C is not shown in the figure), to 1300 °C caused the development of the high temperature form, X2 (PDF#21-1458), maintained also for further temperature increases. Interestingly, the transformation between the X1 and X2 phases of Y-MS appears to be virtually complete after the heat treatment at 1300 °C, since most of strongest peaks attributable to the X1 phase (e.g. that at $2\theta \sim 32.5°$) disappeared above 1200 °C. Some residual Y_2O_3, considering the overlapping of peaks, cannot be excluded even at the highest temperatures (in particular, the not indexed peak at $2\theta \sim 29°$ could be attributed to this phase); however, the pycnometric analysis of powdered samples gave a density of 4.53 ± 0.01 g/cm^3, very close to the theoretical value for yttrium monosilicate (4.44 g/cm^3) [24], therefore suggesting the presence of only a limited content of secondary phases.

The phase evolution of samples corresponding to a Y_2O_3/SiO_2 molar ratio equal to 1 constitutes a basis for understanding the more complicated evolution of samples corresponding to a Y_2O_3/SiO_2 molar ratio equal to 0.5, illustrated by the diffraction patterns reported in Fig. 1b. At 900 °C, Y_2O_3 remained again substantially unreacted. At slightly higher temperatures (1000 °C, not shown here, and 1100 °C) Y-MS, in the X1 form, was again the first silicate phase to be formed while a disilicate polymorph, i.e. α-yttrium disilicate (PDF#38-0223), appeared after heating 1200 °C. The low temperature α-phase is known to be obtained when starting from sol–gel formulations [18,19]: as previously shown for the synthesis of mullite [7,8] the approach of ''nano-filled silicones'' is found to yield similar results than sol–gel, but with a much simpler chemistry. The presence of both monosilicate and disilicate phases was maintained even at higher firing temperatures, after the expected polymorphic transformations: the X2 monosilicate replaced the X1 monosilicate in the 1200–1300 °C temperature range, whereas the γ-yttrium disilicate (PDF#38-0440) replaced the α-phase at 1400 °C. The development of the γ-phase is particularly significant, since this is known as the most stable, and consequently desirable, Y-DS polymorph [17]. Also in this case, due to the similarity of the measured density with the theoretical value expected for γ-phase Y-DS (4.15 ± 0.01 g/cm^3 compared with 4.04 g/cm^3 [17]), the content of secondary phases is reputed to be quite limited.

Both Y-MS and Y-DS are known to be used in coating formulations [14], but Y-DS was preferred for the coating of open-celled foams, shown in Fig. 2a, owing to its higher compatibility in terms of coefficient of thermal expansion with the SiC substrate (the coefficient is 4.84 10^{-6} K^{-1} compared to 4.3–5.4 10^{-6} K^{-1} for SiC [14]). The results here presented are quite preliminary, but demonstrate the great potential of the proposed approach. Fig. 2b shows that a uniform coating was achieved with emulsions after stabilization at 300 °C, oxidation at 600 °C (first cycle) and a first treatment at 1400 °C (second cycle). During the stabilization treatment at 300 °C, it is reasonable to assume that, before the completion of the cross-linking process, an increase of coating continuity is obtained by the melting of the polymeric component. The surface of the coated cell struts is not smooth (see also Fig. 2b), and this could be due to partial melting and oxidation of Si (used as sintering aid for the SiC foams), originating small bubbles under the same coating. If the oxidation of the substrate would need to be limited, one could envision a heat treatment in air at a temperature < 1000 °C, necessary the oxidation of the silicone, followed by heating at higher temperature in inert atmosphere, to form the desired silicate phases. Fibrous micro-crystals,

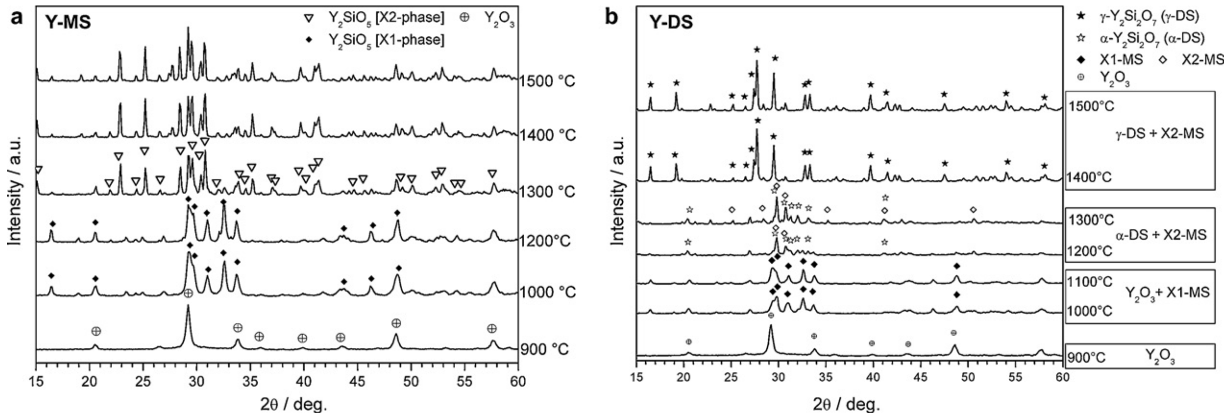

Fig. 1. Phase evolution of silicone-yttria mixtures, for the synthesis of yttrium mono-silicate (a) and yttrium di-silicate (b), with increasing ceramization temperature.

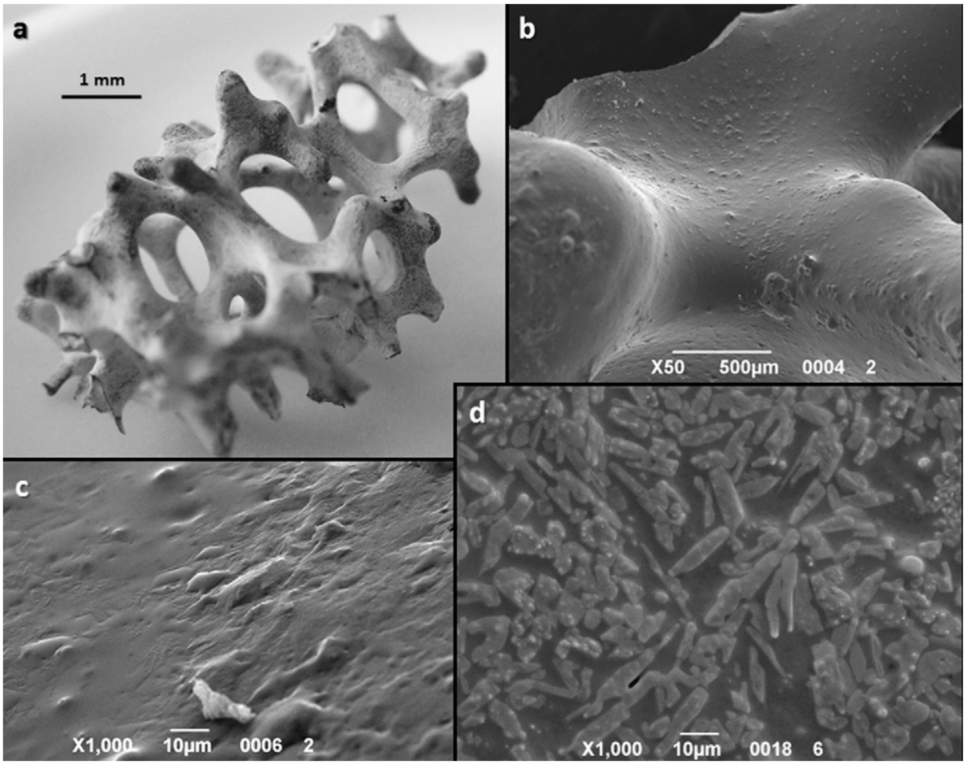

Fig. 2. Details of coated foams: (a) macroscopic appearance; (b and c) strut surface after second cycle; heating at 1400 °C once; (d) strut surface after third cycle; heating at 1400 °C twice.

slightly visible (see Fig. 2c), are the evidence of the starting of the formation of yttrium di-silicate crystals in the coating. After the complete heat treatment, in fact, relatively large fibrous crystals can be observed in the coating (see Fig. 2d, depicting the surface of struts after further depositions and treatment at 1400 °C (third cycle)), and they can be attributed to Y-DS, according to the chemical analysis provided by EDS (energy dispersive X-ray spectroscopy). The surface of the coated strut is very smooth and without any crack; this smoothness could be interpreted on the basis of chemical reactions occurring between the coating and substrate material; in fact, Y-MS impurities in Y-DS are known to interact with silica (from oxidation of SiC and Si) yielding additional Y-DS (Y-MS + $SiO_2 \rightarrow$ Y-DS) [21].

From SEM investigations, the thickness of the coating can be estimated to be of the order of 10 μm. Oxidation tests of the coated foams will be the object of further investigations, although the presence in the foam of hollow struts (reticulated foam) will need to be taken into account when performing the test, as previously observed [23].

As previously discussed, the formulation suitable for forming Y-MS is interesting for a full understanding of the phase evolution for Y-DS. Despite the mismatch in the coefficient of thermal expansion, also the mono-silicate is used for the protection from oxidation of SiC-based materials; some formulations, according to Seifert et al. [15] comprise both Y-silicate phases. The approach of nano-filled silicones could be advantageous, since all intermediate compositions between Y-MS and Y-DS are easily feasible by adjusting the Y_2O_3/silicone weight ratio. Due to the limited availability of Y_2O_3 nano-particles, Y-MS was not tested for the coating of SiC foams (Y-MS is obtained with a higher amount of filler than Y-DS, and coating operations required the usage of relatively large quantity of raw materials in try-and-error tests before finding the procedure previously described); however, Y-MS was chosen for secondary tests, requiring the production of limited quantities of ceramic powders. In fact, Y-MS is well known for an important functional application, in addition to coatings; doped with rare earth ions, Y-MS can be used as a phosphor for LED devices [25–27]. In particular, Y-MS exhibit a red luminescence if doped with trivalent Eu ions (Eu^{3+}), which replace trivalent some of Y ions (Y^{3+}) in the crystal lattice. Y-MS phosphor powders are mainly obtained by sol–gel processing coupled with expensive post treatments (spray pyrolysis, treatment in special atmosphere, flame synthesis) [25–27]: the approach of filled silicones could be a valid alternative, due to its simplicity.

As a preliminary test, Eu_2O_3 nano-particles were added to the silicone resin replacing together 5 mol% of Y_2O_3 nano-particles, i.e. in a relatively high amount (Zhang et al. studied a $(Y_{1-x}Eu_x)_2SiO_5$ system with x varying from 0.005 to 0.05) [26] in order evidence the doping. According to the luminescence spectra reported in Fig. 3a, red luminescence (612 nm) was achieved, after excitation at 394 nm, even for powders fired for 1 h at only 1000 °C. A much more significant emission, however, was visible after heating at 1200 °C. A further firing temperature increase to 1300 °C produced only a weak effect, whereas an increase in firing time (from 1 to 3 h), was very

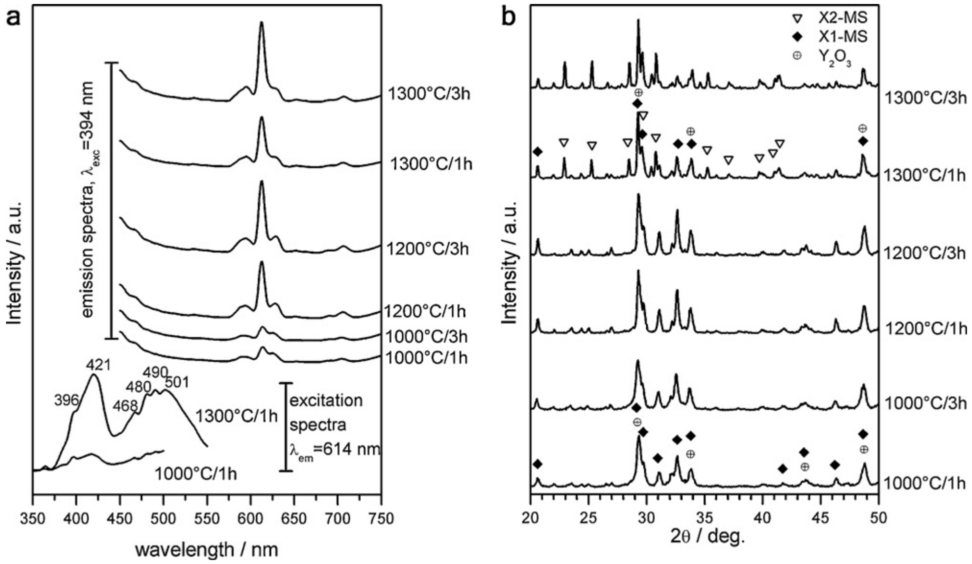

Fig. 3. Photoluminescence spectra (a) and X-ray diffraction patterns (b) of Eu-doped Y-MS, with increasing ceramization temperature and time.

effective, leading to a ~30% increase in intensity, for both heating temperatures of 1200 and 1300 °C.

The activation occurring around 400 nm, i.e. at the boundary between the visible (blue–violet) and the near UV spectrum, is promising for applications in modern LED devices, as many phosphors are coupled with a diode emitting at the wavelength required for the excitation [28], and the red color represents the more important one to be developed for the susceptibility of human eye [27]. The diffraction patterns, shown in Fig. 3b, confirm the achievement of the desired luminescent silicate phases, after only 1 h at the target temperature, with no significant modification of the phase assemblage occurring after heating at 3 h, indicating that the good microstructural stability of the produced material. The increase in firing temperature determined a decrease in the luminescence peak width (Fig. 3a), which can be attributed to the increase in crystal size. Compared to the formulation for pure Y-MS, the conversion from X1 to X2 phase is less complete, the peak at $2\theta \sim 29°$, consistent with both X1 and residual Y_2O_3 being still well recognizable at 1300 °C. This finding may explain the limited improvement observed in the intensity of luminescence passing from 1200 to 1300 °C, which was relatively weak considering that the X2 phase is recognized to be the most efficient phase for emission [27].

Further refinements in the study of luminescent Y-MS ceramics produced from preceramic polymers are needed, especially for precise phase quantification and understanding of the link between phase assemblage and luminescence spectra, with the aim of further optimizing the formulations, enhance the phase purity and possibly the quantum yield. Additionally, it could be interesting to evaluate emissions at other excitation wavelengths; in fact, the excitation spectra in Fig. 3a show that the phosphor may be activated in a relatively wide range of wavelengths. Finally, the flexibility of the approach could be the basis for the development of phosphors with a less or more substantial Eu doping concentration and with a different Si/Y ratio [26,27], arriving even at the composition for Y-DS, much less explored for phosphors [29].

The proposed approach for the production of luminescent materials from preceramic polymers containing nano-sized oxide particles is certainly attractive, because the easy shapeability of the preceramic polymer matrix could be exploited to produce luminescent coatings, or luminescent fibres or luminescent structured (e.g. porous) bodies, in analogy to silicones without fillers [30]. Finally, it should be considered that, upon treatments in nitrogen atmosphere, silicone may lead to a silicon oxycarbide residue (SiOC) [30], instead of silica, with the possibility of carbothermal reaction (already exploited for obtaining SiAlON [31]) and formation of Y-silicon oxynitrides, also particularly interesting as phosphors [32].

4. Conclusions

We may conclude that:

- Silicones containing nano-sized oxide particle fillers have been successfully used for the synthesis of yttrium mono- and di-silicate, produced at relatively low firing temperatures (Y-MS was obtained at only 1000 °C) and for short firing times;
- silicones containing nano-sized oxide particle fillers may constitute valid coating media for the development of an anti-oxidation layer of yttrium-disilicate on the surface of SiC open-celled foams;
- a simple modification in the oxide nano-particle fillers (i.e. partial replacement of Y_2O_3 with Eu_2O_3) led to the synthesis of luminescent Eu-doped yttrium-monosilicate materials. A strong red luminescence was observed in the visible-near UV range, suitable for implementation in modern LED devices.

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