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X International Scientific Conference of Mechanical Engineering

**Cerámica plástica:
procesamiento innovador de parte cerámicas funcionales
*Ceramics from Plastics:
Innovative Ceramic Processing for Functional Parts***

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Abstract:

Abstract: For more than 50 years, research on ceramics has a focus on processing with polymeric precursors. These so-called preceramic polymers consist of an inorganic silicon-containing backbone and organic side groups, which allow for plastic shaping prior to thermal treatment for polymer-to-ceramic transformation, in this paper addressed as pyrolysis, typically under exclusion of air. Due to a massive density change during pyrolysis shrinkage may amount up to 80 vol. %; (partial) compensation can be achieved by addition of particulate fillers.

The combination of the preceramic polymer, the fillers, pyrolysis atmosphere and pyrolysis temperature spans a wide parameter field which allow for a tailor-made microstructure with a multitude of materials functionalities in ceramic processing.

Keywords:

Keywords: *preceramic polymers, polymer derived ceramics*

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1. Introduction

There is a great variety of shaping processes for plastic parts; this holds for primary shaping and forming as well for joining (Franck, 2000), and most of these processes may be applicable to so-called preceramic polymers (Greil, 1995). Preceramic polymers, also addressed as ceramic precursors, are hybrid polymers consisting of an inorganic Si-X backbone – X represents carbon, oxygen, nitrogen or, in some specific cases, boron – with organic side groups or hydrogen as part of the structure-terminating molecular functionalities. The later mentioned make them meltable at elevated temperature or provides solubility in organic solvents, which is a precondition for plastic shaping and/or necessary for a cross-linking processes at low temperatures as part of the shaping (Colombo, Mera, Riedel y Sorarù, 2010).

When preceramic polymers undergo a thermal treatment polymer derived ceramics (PDCs) are formed. PDCs have been in the focus as innovative ceramic processing techniques and materials for ~ 50 years. In comparison to conventional ceramic processing, a significantly wider range of starting materials and process parameters and combinations thereof are possible, resulting in the feasibility of adding functionalities to the materials such as electric conductivity, catalytic activity, tailored surface-chemical properties or low/negative thermal expansion (Barrera, Bruno, Barron y Allan, 2005; Fedorova, Betke y Scheffler, 2017; Fedorova, Hourlier y Scheffler, 2017; Kryukova, Knyazeva, Pogrebenkov, Kostikov y Sevostianov, 2017; Schelm, Schwidder, Samuel, Scheffler y Scheffler, 2017). PDCs are typically processed by means of polymer shaping techniques resulting in a great variety of shapes and parts particularly not achievable by conventional ceramic processing technologies (Melcher, Cromme, Scheffler y Greil, 2003; Reschke y Scheffler, 2012; Woiton et al., 2011; Zeschky et al., 2005) As preceramic polymer precursor materials oxygen-containing (e. g. polysilsesquioxanes) or oxygen-free materials (e. g. polycarbosilanes) may be used. Figure 1 illustrates the most relevant types of preceramic polymers and their corresponding ceramic materials after pyrolysis.

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type of precursor	corresponding ceramic after pyrolysis
polysilsesquioxane $[\text{RSiO}_{1.5}]_n \longrightarrow$	$\text{SiO}_2 + \text{SiC} + \text{C}$
polycarbosilane $[\text{R}_1\text{R}_2\text{SiCH}_2]_n \longrightarrow$	$\text{SiC} (+ \text{C})$
polysilazane $[\text{R}_1\text{R}_2\text{SiNH}]_n \longrightarrow$	$\text{Si}_3\text{N}_4 (+ \text{SiC})$
polyborosilane $[\text{R}_1\text{R}_2\text{SiBR}_3]_n \longrightarrow$	$\text{Si}_3\text{N}_4 + \text{BN}$

Figure 1. Schematic representation of most relevant types of preceramic polymers and their corresponding ceramic materials after pyrolysis.

As a result of thermal treatment a complex set of chemical reactions occur, leading to a polymer-to-ceramic transformation. This complex process is accompanied by a high level of shrinkage, often up to 80 vol. %, or cracks and porosity are formed. In order to minimize the shrinkage effect particulate fillers may be added (Barroso, Li, Bordia y Motz, 2019; Greil, 2000; Torrey et al., 2006). This leads to a reduction in shrinkage up to a total compensation resulting in a zero shrinkage, and near-net shaping is possible (Greil, 2000). An illustration is given in figure 2.

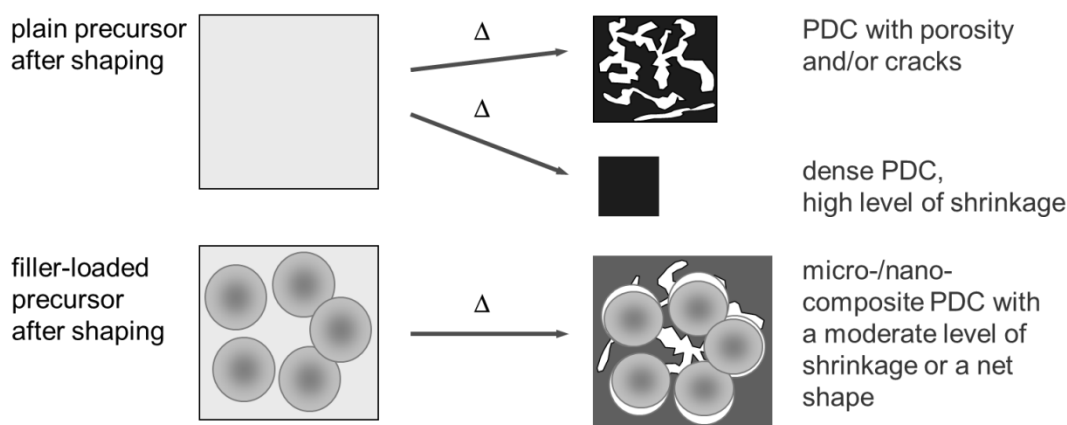


Figure 2. Schematic representation of the microstructure development during the polymer-to-ceramic transformation process without (top) and with (bottom) particulate fillers (adapted from (Greil, 2000)).

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With respect to the reactivity of fillers they are classified into *inert* and *active*: Inert means that the fillers, e. g. alumina, silica, titania, silicon carbide, silicon nitride, do not undergo a chemical reaction with the preceramic polymer or the *in statue nascendi*-PDC. Active fillers such as silicon, titanium, molybdenum, niobium or silicides, react with the preceramic polymer or components of the PDC during PDC formation, and/or with the pyrolysis atmosphere. Thus, active fillers may form new phases within the PDC system, and this is one of the main keys for the generation of additional properties such as electric conductivity, catalytic activity or tailored surface chemical and surface physical properties (Greil, 2000).

2. Major aspects of PDC processing

Shaping and cross-linking. PDC processing starts with the preceramic polymer which is either a powder and still meltable or it is a liquid. Powder processing starts with mixing with fillers. Important is that there is no agglomeration of the fillers or the PDC starting material, and the fillers have a defined particle size, typically in the range of some to some tens of micrometers. For cross-linking a catalyst is to be added and shaping is carried out, e. g. by warm pressing. This was shown to be suitable for low-density as well high-density fillers (Reschke, Bordia, Scheffler y Scheffler, 2016).

Liquid processing is possible with powder-like starting materials when they are soluble in organic solvents such as acetone, isopropanole or monomeric silanes; another liquid processing route makes use of liquid precursors. Advantage of liquid processing is that the viscosity can be adjusted in a wide range, and this is a precondition for the application of different shaping processes. With respect to this, the manufacturing of thin-walled tubes was demonstrated by a centrifugal casting process (Melcher et al., 2003), and tape casting for the manufacturing of high-temperature stable ceramic tapes (Cromme, Scheffler y Greil, 2002), which may be coated with a microporous zeolitic layer for gas separation and catalysis (Scheffler y Scheffler, 2009).

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Pyrolysis and microstructure formation. The pyrolysis process starts roughly from 400 °C and ends between 1,000 °C and 1,400 °C. During pyrolysis the polymer-to-ceramic transformation occurs. Even when there are numerous publications available dealing with chemical mechanisms, a comprehensive description of pyrolysis remains complex. With respect to a broad variety of reactions between the fillers, the components formed during pyrolysis, the pyrolysis atmosphere, the containment, impurities and the like, there is, due to the fact of gas release during pyrolysis, a local change in chemical activity over the distance within a bulk sample. This makes it difficult to generate intact and well-describable compact PDS parts; the chemical composition over the distance within a sample may be heterogeneous. The PDC community, therefore, is focused on small-dimensional parts such as coatings, foams, MEMS and fibers.

One more aspect is the microstructure with respect to crystallinity. Crystallization processes may be triggered by the presence of fillers, which act as heterogeneous seeds. The absence of fillers, on the other hand, may result in an amorphous or nanocrystalline material. A schematic illustration of the thermal steps is given in figure 3. More details related to PDCs may be found in (Colombo, 2010).

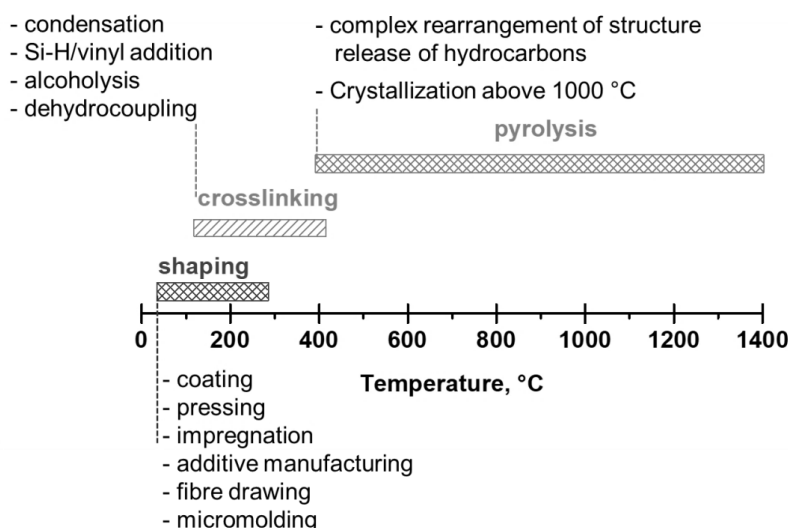


Figure 3. Processes related to the steps shaping, cross-linking and pyrolysis/crystallization, adapted from Colombo et al.

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3. Examples of PDCs for mechanical and process engineering

Oxidation protection coatings for high-temperature alloys. Metal surface protection against overheating and oxidation at high temperature is a challenge. Some of the coating requirements are: Chemical and thermal stability under harsh conditions and high temperature, good adherence on the metal substrate, similar coefficients of thermal expansion, optional repair function when the coating has pores or cracks. The feasibility of coatings from filler-free and filler-loaded preceramic polymers and its thermal conversion was shown for a variety of metal systems. Motz et al. (Günthner, Kraus, Dierdorf et al., 2009; Günthner, Kraus, Krenkel et al., 2009; Günthner, Wang, Bordia y Motz, 2012; Kraus, Günthner, Krenkel y Motz, 2009) demonstrated this with a perhydropolysilazane as the preceramic polymer and a wide range of different fillers. The authors achieved excellent adherence, e.g. on steels, titanium, molybdenum, and the resulting coating thicknesses were between some micrometers in filler-free systems and some hundreds of micrometers in filler-loaded PDC systems; a model for the description of the critical coating thickness was established by Bordia et al. (Torrey y Bordia, 2008). These authors investigated also the expansion behavior of fillers, when pyrolysis was carried out in reactive atmosphere (Torrey y Bordia, 2007).

With the as developed PDC coatings it is possible to protect molybdenum-silicon-boron alloys against molybdenum pesting (Smokovych, Hasemann, Krüger y Scheffler, 2017; Smokovych y Scheffler, 2018a, 2018b), which is an effect that occurs in the temperature range between 600 °C and 800 °C, when the silica layer that typically occurs in this system is not yet formed due to low temperatures (Paswan, Mitra y Roy, 2006). In this temperature range, molybdenum oxidation and evaporation occurs with a dramatic consequence for the entire material: an unprotected alloy loses its mechanical integrity because of a high weight (Mo) loss (Parthasarathy, Mendiratta y Dimiduk, 2002; Perepezko, Rioult y Sakidja, 2008).

Figure 4, left, shows a SEM image of a cross-section of a PDC coated molybdenum substrate. The coating material consists of a 15 wt. % B and 25 wt. % Si filled polysilazane

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which was cross-linked at 110 °C in air and subsequently pyrolyzed at 1,000 °C in nitrogen atmosphere (heating/cooling rates were 3 K min⁻¹). No phase segregation was detected and the material (coating only) was tested at 800 °C in air for 100 h. Figure 4, middle, shows a SEM image of the as received material and the material after the 100-h-test at 800 °C in air is shown in figure 4, right.

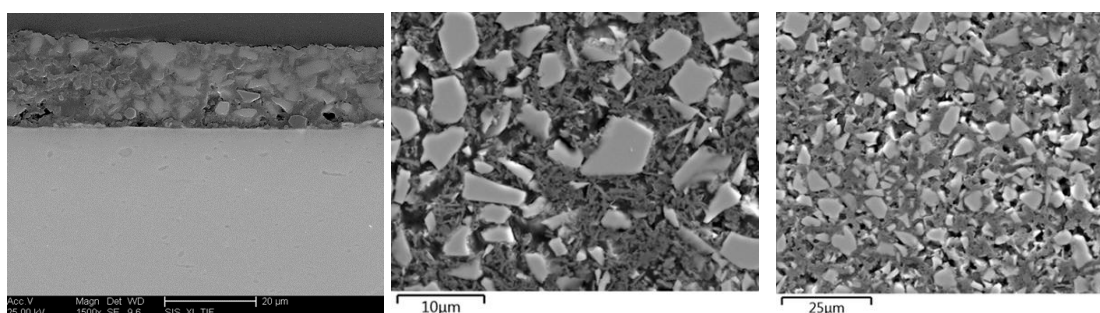


Figure 4. SEM image of a cross section of a PDC coating on Mo after pyrolysis at 1,000 °C in nitrogen atmosphere, left; as received PDC coating material after pyrolysis at 1,000 °C in nitrogen, middle, and PDC material after pyrolysis and 100 hours exposure to air at 800 °C.

No significant difference in the microstructure was detected after a 100-h exposure to air. From this we deduced that this material is suitable to protect Mo based alloys, and, in fact, it has been shown that the weight change takes order to a parabolic law which indicates the formation of a protective SiO₂ layer. In this layer also boron was found indicating the formation of a low viscosity borosilicate glass, which may protect the base metal beneath the oxidation protection coating.

Polymer derived ceramics with tailored surface properties. As described above, PDCs have a high potential in surface tailoring. Because of the polymer-to-ceramic transformation which is accompanied by the change in composition, in particular from organic-dominated surfaces to inorganic-dominated surfaces, they change their physicochemical properties. This means, on the other hand, that their wettability with hydrophilic/hydrophobic liquids may change, and this effect may be useful in liquid multiphase flow phase separation and extraction. In (Schelm et al., 2017) we described

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the influence of the surface chemical properties of ceramic PDC-coated foams on the separation effect of a liquid two-phase system in an extraction column, and it has been shown that the surface energy of the foams effect the effectiveness of the reactive extraction reaction. The foams with different surface chemical properties, varying from hydrophilic to hydrophobic, were used as static mixers in a reactive extraction of an organic dye between two non-miscible liquid phases. In comparison to the extraction without foams the extraction rate between the two liquid phases was significantly. The extraction rate clearly showed an influence of the pyrolysis temperature and thus the contact angle/surface energy of the foams on the separation rate. With increasing pyrolysis temperature and also increasing surface energy and wettability the extraction rate increased. It was supposed that this is a result of a more intensive wetting of foams with high surface area with aqueous phase and so an increased *liquid-liquid* surface area.

Figure 5 shows the foams used for the above-described experimental setup after pyrolysis at different temperatures: with increasing temperature the foams become darker and at least they appear black. This effect originates from the formation of free carbon typically present after higher pyrolysis temperatures.

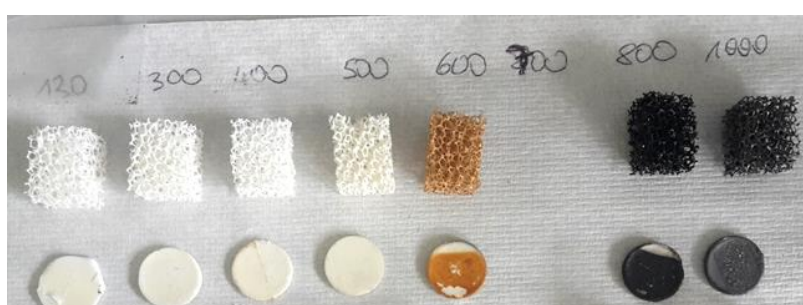


Figure 5. Alumina foams after coating with a polysilsesquioxane-type preceramic polymer and subsequent pyrolysis at temperatures between 120 °C (cross-linking) and 1,000 °C (from left to right).

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The resulting contact angles for water and diiodmethane and surface energies (as measured on planar tapes of the same composition and pyrolysis conditions) are presented in figure 6.

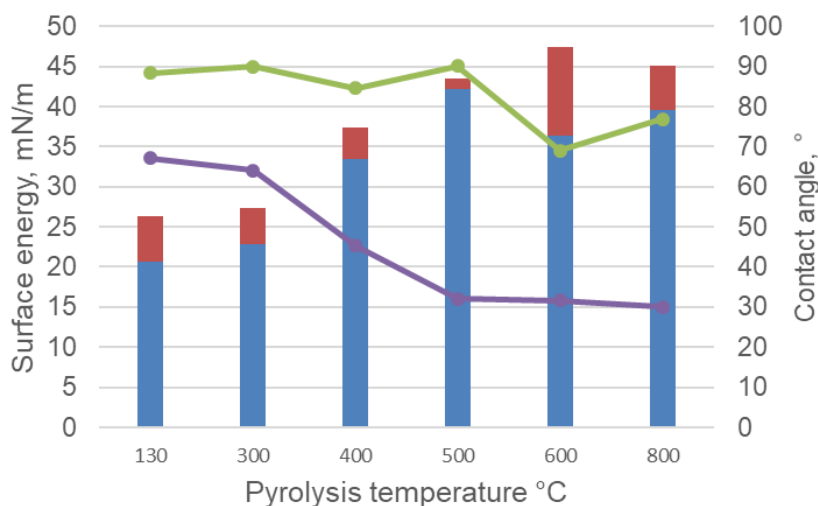


Figure 6. Polar end disperse ratio (red and green bars) and contact angle for water and diiodmethane (green and purple lines) as measured on planar tapes of the same composition as of the PDC coated alumina foam. *red: polar ratio blue: disperse ratio green: water contact angle purple: diiodmethane contact angle*

4. Conclusions

Manufacturing of ceramics with preceramic polymers is a versatile processing route suitable to obtain materials with specific properties. It is possible to functionalize ceramics with a multitude of properties. This was demonstrated in this paper for oxidation protection coatings on molybdenum alloys having the potential for the formation of an oxygen diffusion-limiting glassy layer and, not shown here, with a self healing function. In a second example it was demonstrated, that surface chemical properties can be tailored ranging from hydrophobic to hydrophilic. This might be applicable in process engineering and phase separation, however, a great number of different applications is conceivable.

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