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TECHNOLOGICAL PROPERTIES OF SiC-BASED CERAMIC SLURRIES FOR MANUFACTURING INVESTMENT CASTING SHELL MOULDS

WŁAŚCIWOŚCI TECHNOLOGICZNE MAS LEJNYCH NA BAZIE SiC STOSOWANYCH DO PRODUKCJI CERAMICZNYCH FORM ODLEWNICZYCH

In this work technological properties of ceramic slurries based on silicon carbide were characterized. Silicon carbide and poly(vinyl alcohol) in an amount 6%, 10% and 15 wt. % as a solution were added to prepare the ceramic slurries. Solid phase content in ceramic slurries was 62,5 wt.%. Plate weight test, pH, density, viscosity (measured by the Zhan 4# cup) and dynamic viscosity were investigated. Dipping test on wax model was done to measure adhesive properties. Characterization of SiC fillers powder was made by determination of grain size, density, chemical composition, Zeta potential and by use of scanning electron microscopy. Obtained results of ceramic slurries based on SiC meet standard specification in industrial investment casting and its properties are very promising for future application in investment casting of superalloys.
Keywords: binders, investment casting, poly(vinyl alcohol), SiC slurries, shell moulds

W artykule przedstawiono wyniki badań właściwości technologicznych mas ceramicznych na osnowie SiC mogących znaleźć zastosowanie do produkcji form odlewniczych do odlewania precyzyjnego części turbin lotniczych. Zawiesiny ceramiczne z SiC o stężeniu 62,5% otrzymano w mieszadle mechanicznym z dodatkiem 6, 10, 15% wag. alkoholu poli(winylowego) spełniającego rolę spoiwa oraz upłynniacza. Przeprowadzono szereg badań materiałoznawczych dla proszku, polimeru oraz wytworzonych mas lejnych, w tym lepkość względną i dynamiczną, pomiar gęstości, pH oraz zanurzeniowy test płyty. Pomiary prowadzono przez 96h. Udowodniono, iż właściwości technologiczne oraz reologiczne ceramicznych mas pozwalają na zastosowanie ich do produkcji form odlewniczych przeznaczonych do procesów odlewania precyzyjnego części turbin lotniczych.

1. Introduction

Silicon carbide (SiC) is widely used in various branches of industry, for example in: construction engineering, chemistry, electronics or astronautics. It exhibits advantageous properties like: high strength, hardness and resistance to thermal shocks and oxidation. It is also characterized by thermal resistance and stability as well as very high thermal and electrical conductivity [1]. In comparison with other ceramic materials (like Al_2O_3, $ZrSiO_4$, ZrO_2 or mullite) SiC exhibits the highest thermal conductivity. Therefore SiC is potentially useful for manufacturing ceramic shell moulds. Because due to high thermal conductivity (130 W/mK in 20°C), SiC gives possibilities to control macro and microstructure parameters (like grain size or interdendritic distance) in cast materials. Simultaneously increasing their mechanical strength at elevated temperatures. Additional advantages of silicon carbide are: affordable price and wide availability.

Nowadays investment casting industry in Poland base on binders made from hydrolysed ethyl silicate. Pure ethyl silicate has no binding properties but it is susceptible to hydrolysis in water, what makes it useful in manufacturing ceramics slurries. The water insoluble silicon dioxide (SiO_2) is obtained from ethyl silicate hydrolysis. Silica is characterized by chemical inertness, resistance to high temperatures and capability to bind ceramics powders. Addition of a solvent accelerates the process, but has also a negative impact on the environment and what is more important – it causes work conditions deterioration for moulders [2-3]. In spite of that ceramic slurries based on hydrolysed ethyl silicate have also some advantages. They are characterized by high stability of technological parameters and causes reduction of ceramic layer drying time. Moulds based on this binder exhibit high resistance, high gas permeability and facilitates the ease of knocking out of casts.

Nowadays attention is attracted by binders containing colloidal alumina. These are promising water-based binders, which can be alternative to other polymers used in the investment casting industry. Such binders are characterized by lower melting points, higher thermal conductivity and better dispersion in both aqueous and inorganic solvents. The second aspect of novelty is usage of water-soluble polymers, acting as rheology modifiers and binders provide appropriate strength of ceramic shell moulds [4-5].

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Currently binders which are soluble in alcohol are commonly used in manufacturing ceramic shell moulds, due to their significant properties. Substances based on alcohol have negative influence on the environment, reduce work comfort and are unhealthy for human. This can be prevented by applying low-cost polymeric binders, which are soluble in water. Minimum amount of these binders provides high mechanical strength of shell moulds in raw state. Water-soluble binders used in various moulding processes such as: moulding, casting and receiving thin films ("tape-casting") includes: poly(vinyl alcohol), poly(oxyethylene)glycol, carboxymethyl cellulose and methylcellulose water-based adhesives such as: polyacrylic and polyurethane dispersion [6-13].

Up to now the preparation of shell moulds with poly(vinyl alcohol) are not carried out in aerospace industry. Studies described in this paper are very innovative [14].

Selection of a suitable binder and its quantity is an important issue in fabrication ceramic materials [15-16].

Poly(vinyl alcohol) (PVAL) is a synthetic polymer in form of white or cream dry powder or granules. It is very useful in industry, for example as an additive for textile fibers, adhesives stabilizer and also in cosmetic industry as an ingredient in creams, soaps or toothpastes. In addition due to its high strength, water-solubility and low cost it is one of the most commonly used binder in ceramics technology. Both in cosmetology and ceramics technology PVAL acts as a binder.

The aim of the study was to examine the effect of adding PVAL on the properties of ceramic slurries used in manufacturing SiC shell moulds for investment casting of aircraft turbine engine parts. PVAL was used as liquefier.

2. Experimental methods

Within this studies the SiC-based ceramic slurries were manufactured. They have been formulated using a mixture of: nanoalumina powder, poly(vinyl alcohol) and standard additives like anti-foam and wetting agents (Evonik, Germany). The poly(vinyl alcohol) 26-88 was delivered by Mowiol, Germany. It was characterized by molecular weight 26000 g/mol and hydrolysis degree 88%. PVAL amount in slurries was varied: 6, 10 and 15% by weight.

At first the SiC powder type 99C – green variant (Stanchem Polska) was tested. X-ray fluorescence (XRF) analysis were carried out to measure chemical composition. Bruker S4 Explorer X-ray fluorescent spectrometer was used. The spectrometer was equipped with a Rh tube with a copper anode, Cu, Pb and Al filters, 0.23°, 0.46°, 1°, and 2° collimators, and LiF200, Ge, PET, and XS-55 crystals.

Microstructure of SiC powder was examined using a Hitachi SU-8000 scanning electron microscope. Observations were performed at 5 kV accelerating voltage using a secondary electrons detector. Samples of SiC powder were placed on carbon films.

The next step was to characterize manufactured SiC-based ceramic slurries. Plate weight tests properties were measured using a 75×75 mm brass plate with a weight of 75.46 g. The procedure used in this process has been developed in earlier experimental studies. The dripping effect was observed for 180 s, with results recorded at 15 s intervals. Measurements were taken over 4 days in an air-conditioned lab at 21°C.

Relative viscosity was measured using a Zahn cap with the nozzle diameter of $\varphi = 4$ mm. The pH of tested substances was measured with a pH-meter equipped with a dispersion electrode. The density was determined using an aerometer with a measurement range of up to 2.5 g/cm^3.

Dynamic viscosity was measured with a Brookfield DV II+ (USA) viscometer using the coaxial cylinder method with calibrated spring coupled to a spindle rotating in the dispersion. Rheological properties of the test substances were measured in the ranges of 10÷200 and 200÷10 rpm.

3. Results

Results of XRF chemical composition analysis of silicon carbide powder are shown 99,41 wt.% of Si, 0,323 wt.% of P and 0,263 wt.% of Ca. Type 99C silicon carbide powder is characterized by high purity. Additionally elements such as: P, Ca, Ni and Fe were present in quantities not exceeding 0.6 wt.%. Examples of microstructure images of SiC are presented in Figure 1. SiC particles are sharp-edged and irregularly shaped, which is advantageous in terms of their capability to form smooth wax model surfaces.

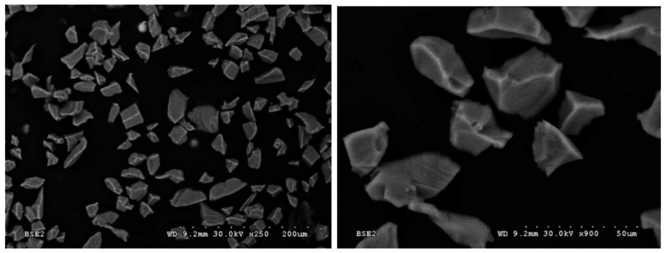

Fig. 1. SEM images of SiC powder designation F400 99C

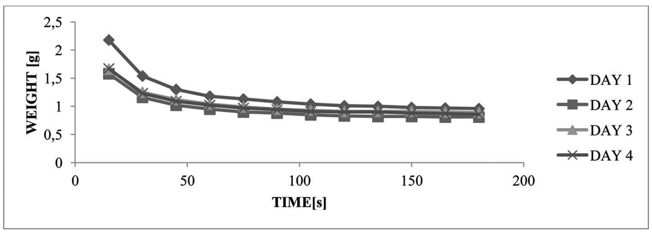

Fig. 2. Plate weight test results of SiC with 6 wt.% of PVAL addition

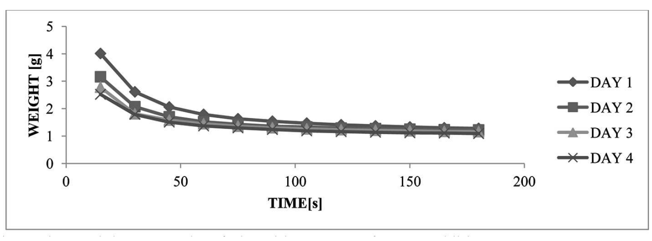

Fig. 3. Plate weight test results of SiC with 10 wt.% of PVAL addition

Figures 2÷4 presents drip test results for ceramic slurries with PVAL addition of 6, 10 and 15% by weight. As seen in Figures 3÷5, the curve profiles are similar. The effect of the PVAL addition led to a initial viscosity reduction of the slurries. This is related with a increase of initial amount of ceramic slurry deposited on the plate weight. After approximately 80s of dripping, a plateau was observed and it was maintained throughout remaining test period in each ceramic slurries.

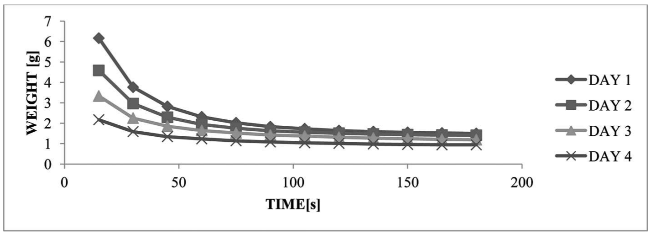

Fig. 4. Plate weight test results of SiC with 15 wt.% of PVAL addition

Images of SiC layers observed upon dripping of the ceramic slurries are shown in Figure 5. Presents photographs illustrating the amount of ceramic slurries deposited on the plate after test. Very good quality of surface coverage can be observed in each case. The powder particles were very adhesive to the slurry on both flat surfaces and edges.

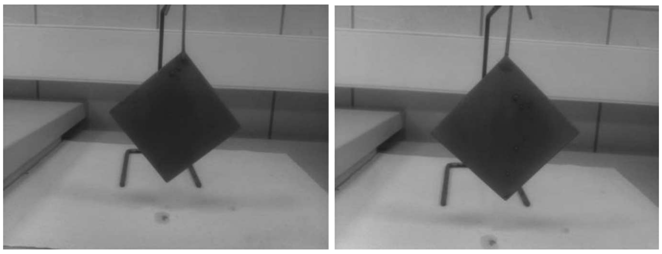

Fig. 5. Plate weight test example photographs of SiC slurries

As seen in Figure 6, ceramic slurries containing 15 wt.% PVAL had the highest viscosity of about 430 mPa*s. Reducing the amount of PVAL leds to a lowest level of dynamic viscosity. For the this slurries dynamic viscosity was about 380 mPa*s for 10 wt.% PVAL and 195 mPa*s for 5 wt.% PVAL.

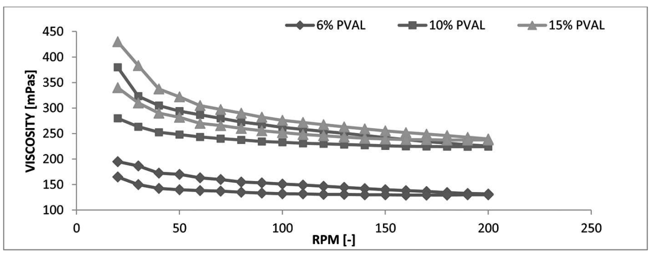

Fig. 6. Dynamic viscosity diagram of SiC with 6, 10, 15 wt.% PVAL addition

Figure 7 presents the distribution of relative plate weight test after 120s and Zahn cup 4# of the ceramic slurries measurement. It shows the influence of adding PVAL on relative distribution between Zahn cup viscosity and plate weight test measurement. It is evident that the relative viscosity of ceramic slurries was different on each day of experiment. For slurries containing 6 wt.% and 10 wt.% of PVAL the cup discharge time decreased with each day of measurement. In the third case (slurries containing 15 wt.% PVAL) the discharge times were increase on the last day of the experiment. This was due to the stabilization of the ceramic slurries over successive days, which was the result of better homogeneity of the tested substance and stable pH of the solution. The increase of the polymer content led to higher value of viscosity and plate weight test.

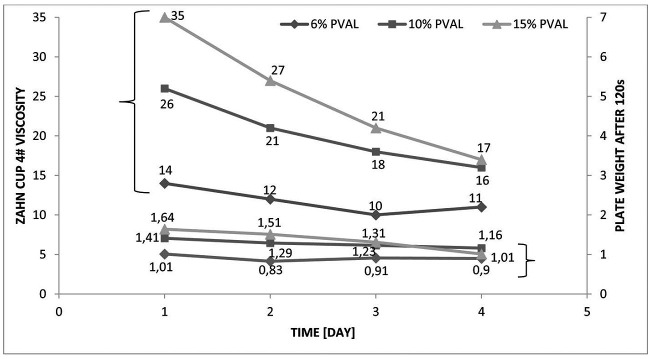

Fig. 7. Diagram of Zhan cup 4# viscosity and plate weight after 120s

Density and pH of ceramic slurries after adding PVAL were stable on each measurement day. These measurements did not show significant fluctuations of density and pH levels.

4. Summary

According to work assumptions, as well as in connection with the industrial requirements, this studies were aimed to develop ceramic slurries, which are characterized by low density, relatively high ceramic powder content and low price. The scientific and technical objectives were to prepare and examine suspensions for manufacture high thermal conductive moulds. Presented studies are one of first available worldwide, which concerns precision casting.

This article presents results of testing ceramic slurries, which consists of a SiC – nano Al_2O_3 matrix, for potential use in manufacturing conductive ceramic moulds for casting aircraft engine parts from nickel superalloys. Al_2O_3 contained in binders was used as a nanofiller suspension and was added into the mix as a polymer dispersion, acting as main binder and diluent. The other binder was poly(vinyl alcohol) (PVAL), which also acted as a deflocculating agent. The effects of PVAL addition were observed mostly by the density and pH reduction in ceramic slurries along with increase of PVAL levels. The highest effects of PVAL were observed in reduction of SiC dispersions viscosity. Addition of wt 6% PVAL solution was insufficient. Ceramic slurries were non-homogeneous, characterized by the lowest viscosity and relatively broad and incomplete hysteresis loop. Increasing the PVAL content to 10% and 15% by weight cause higher viscosity while maintaining the same solid phase level of 62,5% by weight. In those cases obtained hysteresis loops were more narrow and flat. All tested ceramic slurries meet the fundamental industrial criteria for first layers, i.e. viscosity causing the cup discharge times of 25-40 s. However, trials were being conducted to create ceramic slurries with discharge times oscillating around 25 s with the highest possible content of solid phase.

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