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NASA TECHNICAL MEMORANDUM

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TECHNIQUE FOR REMOVING RESIN FROM A MOLDED OBJECT

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION WASHINGTON, DC 20546 MAY 1983

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Application for Patent

1. Name of Invention

Technique for removing resin from a molded object.

2. Range of Patent Application

1. The present invention is a technique for removing resin from a molded object in which the resin is first mixed with a ceramics powder or metal powder. This mixture is then molded and when the resin is removed, it is removed by heat. The molded object is then place into a container which is sealed and large enough to allow the gas from the resin to be controlled by heat from the resin. The gas pressure at the surface of the object is increased by the gas pressure generated from the resin and the resin removed.

2. The present invention is a technique for removing resin from a molded object as described in paragraph 1 above of this Range of Patent Application in which the increase in gas pressure from the surface of the molded object is 1.5 atm to 3 atm at 350°C-400°C.

This invention is a technique for removing resin from a molded object especially from an object in which a resin is mixed with either a ceramic powder or a metal powder. This mixture is then molded into a specified shape by a method such as injection molding. The resin is then extracted from the object derived from molding.

In recent years, the use of caramics has increased and become highly diversified. It is now used in almost every area from general industrial materials to electronics components. Along with this increased use is a demand for much more stringent requirements in regard to precision in dimension, material characteristic and form.

In order to deal with these demands, a number of different methods for producing specific ceramics have been proposed and put into practice, and most consist of adding approprinte types of resins to the ceramic materials. The mixture is then thermoplasticized. The *Numbers in the margin indicate pagination in the foreign text. <u>/1</u>*

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material is then molded by injection molding techniques. Then the resin is removed by thermal cracking methods. After that, the object is fired.

An important point in those techniques is that there must, of course, be no cracks in the object during injection molding, but, just as importantly, the operation of removing the resin must be performed so no cracks or expansion occurs in the molded object.

In conventional methods of resin removal, cracks and expansion are prevented by slowly removing the resin over a period of several tens of hours or a hundred hours in air, and at the same time, raising the rate of temperature increase per time unit in extremely minute increments.

However, techniques like this obviously require long periods of time, hardly satisfactory for industrial use. Even then, we have no assurance that cracks and expansion will be prevented.

The inventors of the present technique took those points into consideration and as a result of many hours of research and many different tests we discovered methods which were industrially feasible and would remove resins without any actual generation of cracks or expansion. We found a method which fulfills the above objectives. We removed the resin from the molded object by heating the object in a sealed container large enough so that the heat can be used to control the pressurized gas. The gas generated from the resin increases the pressure of the gas coming from the surface of the molded object.

Strictly speaking, the size of the sealed container used in this invention differs somewhat according to the form of the molded object and the type of resin used. However, generally, about half of the resin's weight in the molded object will be gassified, and removed, at 350°C - 400°C. An appropriate size is one that can accommodate from 1.5 atm to 3 atm when the container is full.

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If the pressure is not up to this range, that is to say, if the container is too large, cracks and expansion will be produced and the objectives of this invention will not be met. On the other hand, if the pressure is greater than the above cited range, removing the resin from the molded object will be a difficult and extremely time consuming task.

The seal for the container used in the invention does not have to be perfectly tight. Slight pressure leakage causes no problem as long as the above mentioned pressure range is approximately maintained.

The basic material for molded objects described by this invention could be any of the standardly used ceramic powders or metal powders. Examples of usable ceramic powders are: silicon nitride, alumina, <u>kojeraito</u>, zirconia, silicon carbide, aluminum nitride, and tungsten carbide. Some examples of the metal powders are: silicon, titanium, and zirconium.

There are no particular restrictions on techniques for molding the object, but, the best are injection and extrusion molding or any technique that fits those two categories.

Examples of resins which can be used in the invented technique are polypropylene, polyethylene, polysterene and acrylonytrile butadiene styrene copolymer. Other additives or resins suitable for use in this technique are the well known thermoplastics or lubricants such as diethyl phthalate, dioctyl phthalate, stearic acid and lead stearate.

There are also no particular restrictions on the shape of the vessel or container used or on the material from which it is made. Any material, such as stainless steel, alumina- or boric silicate glass, which does not react with the resin at high temperatures is suitable. /5

In actually removing resir from the molded object according to the technique provided for by this invention, the molded object is placed inside the container and heat is applied from the exterior by a suitable heating method.

When using the technique provided by this invention, the temperature is increased at a rate of $5^{\circ}C - 6^{\circ}C$ per hour in contrast to the $3^{\circ} - 4^{\circ}C$ temperature increase rate of conventional techniques. This means that the new technique can remove the resin from the molded object in about half the time conventional methods require. Moreover, the generation of cracks, swelling and expansion is greatly reduced.

We will next explain the technique using an actual prototype example.

Prototype Example

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A mixture of silicon nitride 77.6% by weight, polystyrene 10.6% by weight, polypropylene 5.9% by weight, polyethylene 2.8% by weight, diethyl phthalate 1.5% by weight, and stearic acid 1.5% by weight was heated at a temperature of 250°C. The mixture was then injection molded at a die temperature of 50°C and an injection pressure of 1 ton/cm². This resulted in a molded object 60mm x 100mm.

The molded object was then placed into a 150cc stainless steel container with a screw-on lid. Using an electric thermal convection oven as the heat source, the molded object was heated from room temperature to 200°C at a heat increase rate of 30°C per hour. The rate of increase was changed to 5°C per hour and the temperature raised from 200°C to 350°C. Temperature was then maintained at 350°C for 10 hours. The molded object was then removed from the oven and absolutely no cracks or expansion was observed. The molded object was then fired in a nitrogen atmosphere with the heat raised from room temperature to 1750°C at a rate of 200°C per hour and then maintained at 1750°C for one hour. The resultant sintered object showed no evidence of cracks or swelling.

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Representing the Applicant Patent Attorney Kenji Motohashi and Associate

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