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LABORATORY EQUIPMENT FOR HOT PRESSING

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A laboratory equipment, allowing high-density ceramic materials to be prepared by sintering powders at high temperature under simultaneous effects of axial pressure of up to 40 MPa, either in an inert atmosphere or in vacuo, was constructed. The specimens 15 mm in diameter and 7 mm in height can be utilized as semi-products for the manufacture of cutting tools, components of bearings, etc.

INTRODUCTION

Modern types of ceramics based on nitrides, carbides, borides, etc. are finding ever expanding fields of application particularly in engineering, owing to their advantageous mechanical, physical and chemical properties. Nowadays, use is made of three basic methods in the production of engineering ceramics by the sintering of powders having suitable properties: They are the so-called free sintering, hot pressing and hot isostatic pressing. The respective demands on produtions technology increase in the given sequence, but so does the quality of the final product [1—4].

Hot pressing is a method allowing high-density products to be obtained under simultaneous effects of high temperature and axial pressure. The use of pressure permits the amount of sintering aids to be reduced: as these substances form a liquid phase in the course of sintering during the free process, their partial elimination has a favourable effect on the final properties of the product [2,5]. The hot pressing method is particularly advantageous for the production of simple shapes such as cutting tools, wire drawing dies, components of bearings, and the like. The equipment described below was designed for purposes of basic research in the field of engineering ceramics, in the Laboratory of Sintered Materials at the Slovak Academy of Sciences.

DESCRIPTION OF THE HOT PRESS

The hot pressing equipment, shown in Fig. 1, consists of a furnace, a lever press, a dilatometer and a power source. A schematic diagram of the furnace is given in in Fig. 2. The furnace jacket *6* and its flanges, which also serve as power supply leads, are cooled with water. The jacket is also provided with exhausting and gas outlet and inlet fittings. Graphite meander element *1* was used for heating, surrounded by graphite shields *2* and graphite felt insulation *3*, again covered with a molybdenum shield *4* which separates the outer thermal insulation of Sibral-Super ceramic wool *5*.

Compared to the classical tubular heating elements, the graphite meander with an adjusted cross section (Fig. 3) has several advantages. First of all, it exhibits a longer zone of homogeneous temperature owing to the adjusted sectional area of the resistance track, thus partially compensating for the temperature gradient. The meander element allows for a greater variability of dimensions and therefore also of the total resistance in terms of resistivity ϱ_g of the given graphite, which in turn is limited by the maximum admissible power supply I_{max}, or the power source parameters. Values of I_{max} of up to 1000 A can be regarded suitable. Higher currents bring about problems with contact resistance between the element and the leads, and with cooling the latter. There is also the important advantage that thermal expansion of graphite need not be taken into account when using the meander element. The heating element employed was made of graphite EK 499 (Ringsdorff, FRG). Its basic parameters are specified in Table I. The cross section of the element was adjusted by the trial-and-error method because calculation of the cross section for a certain thermal gradient is affected by several variable parameters and was not practicable in the present case (the tempe-

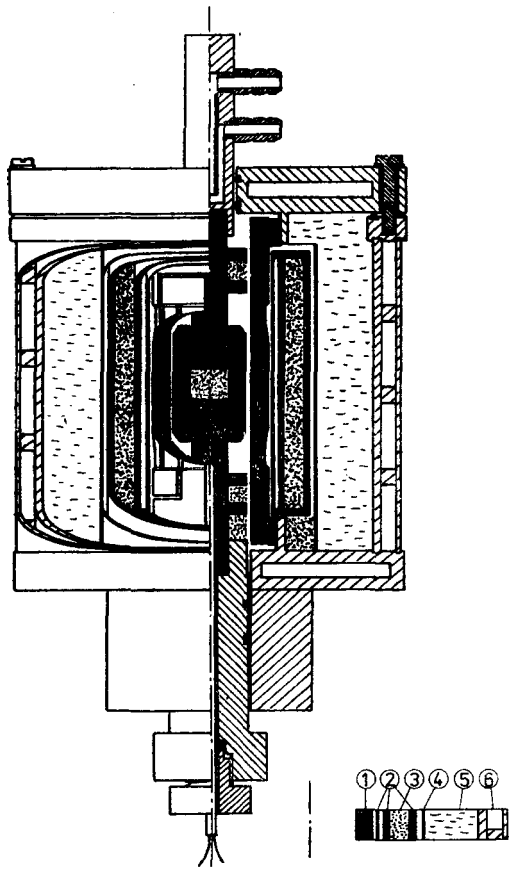

Fig. 2. Schematic sectional view of the furnace part.

Table I
Specification of the graphite heating element

Material	EK 499*
Resistivity, ϱ_g	approx. 9.10 Ohm cm
Total resistance R_g	approx. 10 mOhm
Inner diameter, Φ_i	30 mm
Outer diameter, Φ_i	39—40 mm
Hot zone length, l_h	80 mm
Height of the element, l_a	130 mm

* Ringsdorff, FRG

rature gradient was influenced by the anisotropy of graphite ϱ_g, the temperature dependence of insulation lag, the changes in the thermal conductivity of the inert atmosphere in terms of its pressure, etc).

The contact surfaces between the element and the flanges were coated with a silver paste.

The working space accommodated the floating double-acting graphite die (Fig. 3) with $\Phi_i = 17$ mm and $\Phi_0 = 28$ mm (inner and outer diameter respectively) and 48 mm in height. The mould was of GRAPH-I-TITE material (General Electric, USA), exhibiting a high strength and allowing a relatively large Φ_i/Φ_0 to be used.

The specimen in the graphite die is compressed by graphite pistons of the same material, fitted in pistons of high-temperature steel. Those are guided axially along the frame and sealed in the furnace flanges by o-rings of silicone rubber. A WRe5-WRe20 thermocouple in a corundum double-hole tube, protected by a boron nitride sheath in its hot zone, is placed in the bottom piston. The thermocouple was calibrated by means of standard B. The bottom piston, which brings the specimen into the working space, remains then stationary while the water-cooled upper piston transmits the pressure from the lever press.

The simple lever press has the advantage of exerting a constant uniform pressure onto the specimen in the course of sintering without having to make up the shrinkage. However, the arrangement does not allow the pressure to be changed steplessly during the sintering process. The leverage ratio of the press was 1 : 1.57 or 1 : 4.1. The maximum pressure bearing on the specimen was 40 MPa.

The equipment includes a dilatometer allowing the kinetics of the sintering process to be directly followed [6]. It consists of a mechanism monitoring the upper piston movement, and induction position sensor and a recorder. The range of changes in length is 10 mm without screw adjustment, the maximum sensitivity being 2 μm.

The equipment is supplied by a power transformer whose primary winding is controlled by the EUROTHERM 818P regulator using the NOCONTA AC thyristor device. The relative accuracy of isothermic dwell control is $\pm$ 1 K.

EXPERIMENTAL

The basic specifications of the equipment are listed in Table II. The temperature profile of the working space was measured on a special specimen provided with a hole to allow the thermocouple to be shifted along the specimen. The tem-

perature profile was measured at an isothermal heating temperature of 2100 K. The temperature course is plotted in Fig. 4. The specimen was situated at the point of maximum temperature which was higher by about 15 K than that indicated by the thermocouple during the experiment. The temperature gradient along the specimen was 5 to 10 K in dependence on the specimen height.

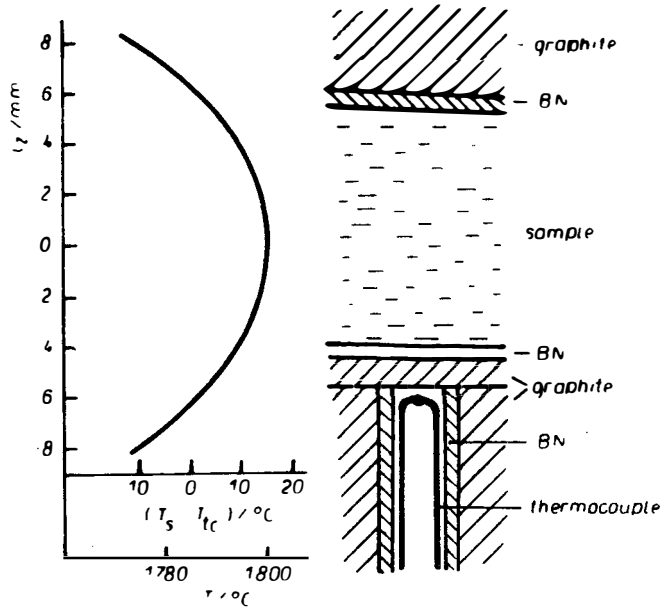

Fig. 4. Temperature gradient along the specimen at 2100 °C
T_s — temperature along the specimen
T_{tc} — temperature measured by the thermocouple.

Table II
Basic specifications of the equipment

Temperatue, T_{max}	2800 K
Pressure, p_{max}	40 MPa
Power demand, P_{max}	approx. 6 kV
Maximum heating rate, r_t	300 K min^{-1}
Specimen diameter, Φ_s	15 mm
Specimen height, l_s	15 mm*

* before shrinkage

The performance of the hot press was verified on three powdered mixtures based on Si_3N_4, listed in Table III. One contained an addition of Al_2O_3 : Y_2O_3 (H8), the second additions of Al_2O_3 : Y_2O_3 and AlN, whose amount was calculated so as to produce β'- SIALON (S8) on sintering, jointly with the SiO_2 present in Si_3N_4. The third mixture (S8C) also contained powdered SiC. The powdered mixtures were prepared by mixing the respective amounts of the components. These

Table III
Composition of the powdered mixtures (wt. %)

Specimen	Si_3N_4	Al_2O_3	Y_2O_3	AlN	SiC
HB	92.00	2.49	5.51	—	—
SB	88.30	2.49	5.51	3.70	—
S8C	84.50	2.49	5.51	3.50	4.00

Si_3N_4 — H. C. Starck H 1 (1.6 wt. % O_2)
Al_2O_3 — Fluka AG (99.99 %)
Y_2O_3 — Lachema Brno (99.99 %)
AlN — Alfa (99 %)
SiC — Alfa (99.8 %)

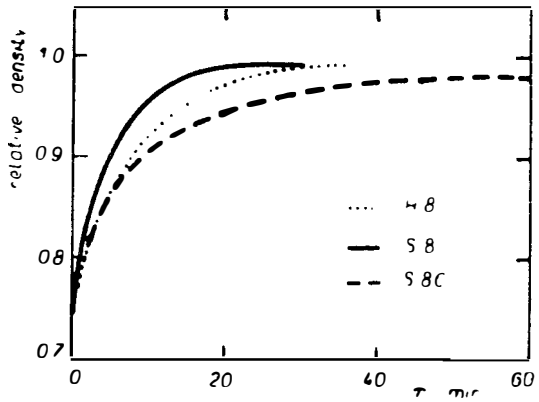

Fig. 5. The course of density vs. the time of isothermic dwell.

were homogenized for 24 hours in absolute ethanol. Following drying and light grinding, the mixes were compacted under a pressure of 100 Pa into pellets 15 mm in diameter and about 12 mm in height. The pellets, protected by a layer of hexagonal BN (99.3%), were placed in the graphite die and sintered in protective nitrogen atmosphere. The heating rate was 200 K min^{-1} and the time of isothermic dwell at 2020 K was 30 to 60 minutes in dependence on the course of shrinkage. The pressure was applied on attaining the temperature of about 1900 K, when the specimen already contained a liquid phase. The shrinkage curves obtained are shown in Fig. 5 as plots of relative density vs. isothermic time of dwell. The diagram demonstates the effect of the various additions on the rate of compacting. One can see that specimen H8 was densified at a much lower rate than S8, obviously owing to different amounts of the liquid phase and different mechanisms involved in the formation of β-Si_3N_4 and β'-SIALON. The addition of SiC likewise slowed down the course of compaction, as it behaved as an inert material in the first approximation; this is indicated by curves S8 and S8C which show that SiC did not take part in the material transport.

CONCLUSION

The equipment described allows fully dense non-oxide ceramics to be prepared under simultaneous effects of temperature of up to 2500 K and pressure of up to 40 MPa in an inert atmosphere or in vacuo. The maximum possible heating rate is 300 K min^{-1}. The working space diameter is 30 mm and the hot zone is 80 mm in length. The temperature gradient along the specimen is 5 to 10 K. The equipment includes a dilatometer exhibiting a maximum sensitivity of 2 μm and permitting the kinetics of sintering to be determined. The size of the specimens, up to 15 mm in diameter and 7 mm in height (after shrinkage) allows the equipment to be used directly in the manufacture of semiproducts for the manufacture of cutting tools, bearing components, an the like.

The equipment was awarded the third prize in the competition of instruments and apparatus developed at the institutes of the Czechoslovak Academy of Sciences and the Slovak Academy of Sciences.

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LABORATÓRNE ZARIADENIE NA HORÚCE LISOVANIE

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Skonštruované laboratórne zariadanie na horúce lisovanie umožňuje pripravu hutnýcn keramických materiálov spekaním práškov za súčasného pôsobenia teploty od 2500 K a axiálneho tlaku do 40 MPa v inertnej atmosfére alebo vo vákuu.

Zariadenie pozostáva z pecnej časti umiestnenej v jednoduchom pákovom lise, dilatometra a napájacieho zdroja. Plášť pece a príruby, ktoré súčasne slúžia ako elektrické prívody sú chladené vodou. Ako výhrevné teleso je použitý grafitový meandrový element. Okolo neho sú grafitové tienenia a izolácia z grafitovej vaty a vaty Sibral-super. V pracovnom priestore je umiestená grafitová lisovacia forma so vzorkou a grafitovými piestami na ktoré pôsobí silou pákový lis. Súčasťou zariadenia je indukční snímač polohy, ktorý umožňuje sledovať zmraštenie vzorky počas spekania. Teplota je meraná termočlánkom WRe5—WRe20 umiestneným v spodnom pieste. Zaradenie je napájané z výkonového transformátora, ktorého primárna časť je riadená programovateľným regulátorom EUROTHERM.

Vzorky o priemere 15 mm a výške 7 mm je možné použiť ako polotovary pre výrobu rezných doštičiek, súčasti ložísk a pod.

Obr. 1. Laboratórne zariadenie na horúce lisovanie.

Obr. 2. Schematický rez pecnou časťou zariadenia (diagramatic view on section of the furnace).

Obr. 3. Grafitovy meandrový element a forma s piestami.

Obr. 4. Teplotný spád pozdĺž vzorku pri teplote 2100 °C.
T_s — teplota pozdĺž vzorky
T_{tc} — teplota meraná termočlánkom.

Obr. 5. Závislosť priebehu hutnosti od času izotermickej výdrže.

ЛАБОРАТОРНАЯ УСТАНОВКА ДЛЯ ГОРЯЧЕГО ПРЕССОВАНИЯ

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Собранная лабораторная установка для горячего прессования предназначена для приготовления твердых керамических материалов спеканием порошков при одновременном действии температуры до 2500 К и аксиального давления до 40 МПа в инертной среде или во вакууме.

Установка состоит из части печи, размещенной в несложном рычажном прессе, дилатометра и источника питания. Рубашка печи и фланцы, служащие для подвода электрической энергии, охлаждаются водой. В качестве нагревательного тела применяется графитовый меандровый элемент. Вблизи него размещены графитовые экранирования и изоляция из графитовой ваты и ваты Sibral-super. В рабочем пространстве помещена графитовая пресс-форма с пробой и с графитовыми поршнями, на которые действует силой рычажный пресс. Составной частью установки является индуктивный датчик положения, с помощью которого можно устанавливать общую усадку во время спекания. Температура измеряется термоэлементом WRe 5—WRe 20, находящимся в нижнем порщне. Установка питается трансформатором высокой мощности, первичная часть которого управляется программируемым регулятором EUROTHERM.

Пробы сечением 15 мм и высотой 7 мм можно использовать в качестве заготовки, предназначенной для производства режущих пластинок, составных частей подшипников и т. д.

Рис. 1. Лабораторная установка для горячего прессования.

Рис. 2. Схематическое сечение частью установки с печью.

Рис. 3. Графитовый меандровый элемент и форма с порщнями.

Рис. 4. Температурное падение вдоль пробы при температуре 2 100 °C, T_s — температура вдоль пробы, T_{tc} — температура, измеряемая термоэлементом.

Рис. 5. Зависимость хода плотности от времени изотермической выдержки.

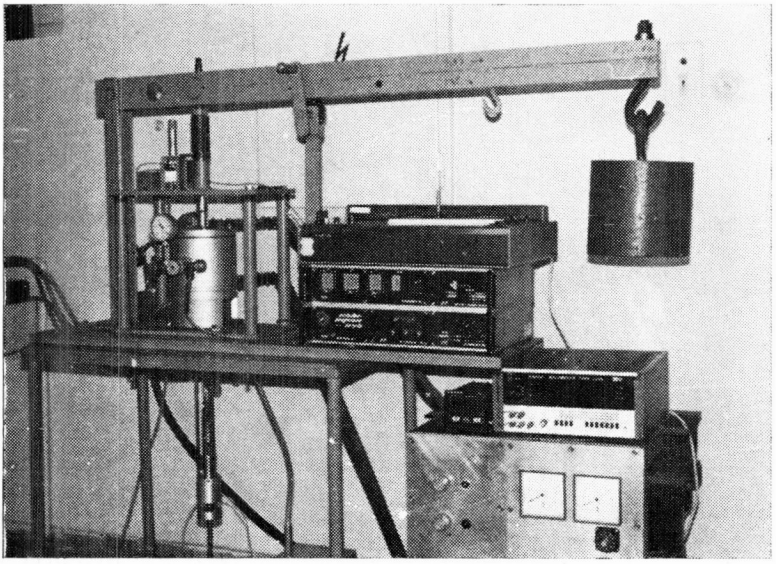

Fig. 1. Laboratory equipment for hot pressing.

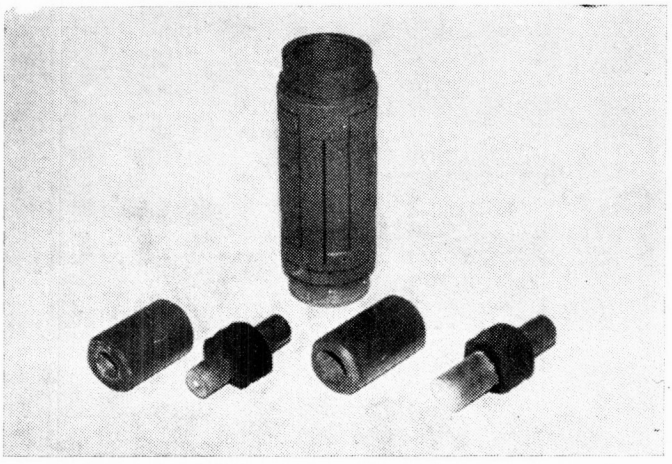

Fig. 3. Graphite meander element and the die with pistons.