N. M. POLONEITCHIK, A. V. KURAKEVICH

INVESTMENT MATERIALS APPLIED IN DENTISTRY



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МИНИСТЕРСТВО ЗДРАВООХРАНЕНИЯ РЕСПУБЛИКИ БЕЛАРУСЬ
БЕЛОРУССКИЙ ГОСУДАРСТВЕННЫЙ МЕДИЦИНСКИЙ УНИВЕРСИТЕТ
КАФЕДРА ОБЩЕЙ СТОМАТОЛОГИИ

Н. М. Полонейчик, А. В. Куракевич

ФОРМОВОЧНЫЕ МАТЕРИАЛЫ, ПРИМЕНЯЕМЫЕ В СТОМАТОЛОГИИ

INVESTMENT MATERIALS APPLIED IN DENTISTRY

Учебно-методическоепособие

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Содержит необходимые студенту сведения о формовочных материалах, применяемых в стоматологии, а также включает основные данные о технологических процессах изготовления зубных протезов, в ходе которых используются формовочные материалы.

Предназначено для студентов 1–2-го курсов медицинского факультета иностранных учащихся, обучающихся на английском языке.

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INTRODUCTION

Technological process of products manufacturing by filling of previously prepared forms (moulds) or by plastic deformation of material in accordance with prepared forms is called **investing**. Polymer and refractory materials, as well as metal alloys, ceramic materials and hemihydrate plaster are used for filling of previously prepared moulds. Metal alloys and thermoplastic materials are subjected to plastic deformation during the investment procedure.

Auxiliary materials called ***investment materials*** are used in dentistry for the production of moulds. Classification of investment materials depending on the final goal of their use (technological processes being used) is presented in the Table 1.

Table 1

Classification of investment materials depending on the final goal of their application

Technological process							
Duplication of gypsum models	**Investment of resins**			**Casting of metal alloys**		**Ceramic injection molding**	**Processing of metals by pressure**
	by investment	**by pressing**	**with vacuum**	**fusible**	**constructive**		
Agar-agar hydrocolloids or silicone materials (A-type, VPS)	Silicone materials (A-type, VPS)	Calcium sulfate hemihydrate			Refractory		Fusible metal alloys
Investment materials							

DUPLICATION OF GYPSUM MODELS

Technologies of model casting of constructive metal alloys, injection molding of ceramics and layer-by-layer sintering of ceramic slip require production of duplicates of working models or of their fragments (dies) made of refractory materials. Thermo-injection investment of resins involves production of working gypsum models duplicates. Technological process of accurate reproduction of working gypsum model in a prototype is called ***duplication***.

Reversible agar-agar hydrocolloids and irreversible vinylpolysiloxanes (VPS, silicone materials of addition type, of additive type of setting) are used for production of moulds during the procedure of duplication.

Materials for duplication of models must meet the following requirements:

- high elasticity, allowing elimination of models from a mould and ability to recover after deformation as completely as possible;
- tensile strength not less than 900 g/cm^2;

− has to reproduce fine details of gypsum models with an accuracy of at least 20 μm;

− minimal shrinkage and high dimensional stability;

− should be nonreactive towards gypsum, refractory materials and modelling materials;

− Melting point of reversible materials should be not more than 100 °C, and in order not to melt the isolating modelling materials on gypsum model working temperature of sol with preservation of its flowability should range from 48–52 °C.

The main component of reversible hydrocolloids is agar-agar (from the Malayan *agar — jelly*). The material is a mixture of polysaccharides of agarose and agaropectin, obtained by extraction from red and brown algae that grow in Black Sea, White Sea and Pacific Ocean. When mixed with water agar-agar forms hydrocolloid. Agar-agar hydrocolloids for duplication of models consist of 3.5 % agar-agar, 35–45 % water and 50–60 % ethylene glycol (O. I. Kruglyakov, G. P. Sosnin, G. D. Obydennov, 1970). Gel is zolated (liquefied) in the temperature range from 70–100 °C and turns again into gel in the temperature range from 30–40 °C. Hydroxyl (OH) groups are able to form hydrogen bonds, which form the structure of gel. During heating these hydrogen bonds are destroyed, spiral structure is straightened, and gel turns into a viscous liquid. This process is reversible and proceeds according to the scheme presented below:

Gel → *heating* **Sol →** *cooling* **Gel**

Several types of agar-agar hydrocolloids with different hardness are produced for duplication of models. The materials are conditionally one-component (fig. 1) and are subjected to heating on a water bath until they become fluid. Before melting it is necessary to cut gel into pieces in order to ensure uniform heating. At a temperature of 70–95 degrees the agar mass on a water bath turns into a viscous liquid, which is cooled to a temperature of 37–42 degrees and poured into a flask.

Fig. 1. Form of industrial production of agar-agar hydrocolloid for duplication of models

Hydrocolloids are softened using microprocessor devices (fig. 2), which regulate heating temperature, and provide maintenance of the working temperature.

Fig. 2. Device for zolation of agar-agar hydrocolloids

Vinylpolysiloxane materials for duplication of models are based on polysiloxane polymer with vinyl groups present at the ends of the macromolecules. The polymerization reaction proceeds via an addition type of reaction due to interaction of a platinum catalyst and a hydrogen-containing silanol. Two-component vinylpolysiloxane materials (fig. 3) are produced in the form of basic component (polyvinylsiloxane, silanol and filler) and catalyst (polyvinylsiloxane, platinum catalyst and filler).

Fig. 3. Two-component vinylpolysiloxane materials for duplication of models

Vinylpolysiloxane materials are dosed in a ratio of 1:1 and mixed manually using a container and a spatula (fig. 4, *a*). Mixing is continued until a completely uniform colour is obtained. Precise dosing, homogenous consistency and elimination of air bubbles from the material can be obtained by automatic mixing using special mixing devices (fig. 4, *b*).

a ***b***

Fig. 4. Container and spatula for manual mixing of vinylpolysiloxane materials (*a*) and device for automatic mixing of components of vinylpolysiloxane materials for duplication (*b*)

Duplication of gypsum models is performed using a demountable container called flask (fig. 5), intended for production of a mould using materials for duplication.

Fig. 5. Flasks for duplication of gypsum models

Schematic representation of the sequence of gypsum model duplication is presented in figure 6. Gypsum model prepared for duplication is installed on the bottom of the flask (fig. 6, *a*). Height of the plaster model base is cut with a trimmer to 1.5 cm. Lateral surfaces of the gypsum model base are made perpendicular to the bottom. Flask base is connected with the body. The flask is filled through the openings located on the top of the body (fig. 6, *b*). After setting of the material for duplication, the base of the flask is removed and the plaster model is eliminated (fig. 6, *c*). The mould (negative reflection) of the gypsum model obtained from the material for duplication (fig. 6, *d*) is filled with the material necessary for production of the prototype model (fig. 6, *e*). After hardening of the material of the prototype model, it is removed from the mould (fig. 6, *f*).

Since the duplication technique may differ depending on the materials used, it is necessary to detail some of the features.

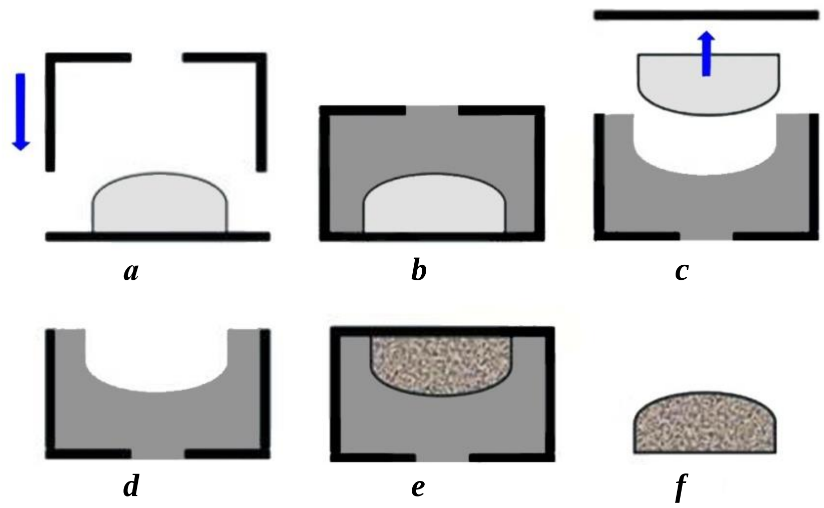

Fig. 6. Schematic representation of the sequence of gypsum model duplication with obtaining of a prototype model

DUPLICATION OF MODELS WITH HYDROCOLLOID MATERIALS

When using hydrocolloid materials for duplication, it is recommended to pre-hold the working model in water for 15–20 minutes at a temperature of 38 °C until the bubbles on the gypsum surface disappear. Exposition of gypsum model in warm water ensures its accelerated saturation and excludes fast curing of hydrocolloid in contact with the «warm» model.

Gypsum model prepared for duplication is installed on a pallet (flask base). Soft wax or play dough can be used for fixation of the model to the base. The pallet is connected with the body, and the flask is filled with the material for the model duplication prepared in accordance with the instruction through the openings located on the top of the flask. Liquid mass for duplication should slowly flow into one of the openings. When the flask is filled, the duplicating mass fills the entire body until the excessive material comes out from all the openings. Cooling of the filled flask is carried out sequentially: in air (at room temperature) for 20–30 minutes or during time period recommended by the manufacturer, and then under running water (at a temperature of 8–10 °C) for the next 30–45 min. After setting of the material for duplication, the pallet is removed and the plaster model is eliminated. After visual assessment of the original model reflection quality in the hydrocolloid material, it is filled with the material for production of the duplicate model. After hardening of the refractory mass, the model is removed from the mould. There is a lot of moisture in the refractory model produced in the agar-agar mass for duplication. Therefore, the hardened refractory model is removed from the agar-agar mould and placed into a drying cabinet. Drying is performed at a temperature of 180–200 °C for 30 minutes. For fixation of the dried model and obtaining smoothness of its surface, the model is immersed for a few seconds into a container with a special hardening liquid, after which the model is re-dried in a drying cabinet or muffle furnace for 10 minutes.

DUPLICATION OF MODELS WITH SILICONE MATERIALS

When using flowable silicone materials, there is no need to pre-soak the gypsum model. During production of a mould it is allowed to apply previously used silicone by putting its pieces around the perimeter of the model base. This allows saving up to 25 % of the material. When using silicone duplicating materials, gypsum model is easily eliminated from silicone using compressed air without damage. The surface of the silicone duplicating mass is coated with a special liquid to eliminate water repellent effect of the silicone surface and is thoroughly dried with compressed air. After eliminating the refractory model from the silicone, it is dried for 20 minutes at a temperature of 80 °C, after which it is necessary to use a special varnish — model fixing agent.

When choosing the material for duplication of models, it should be considered that the advantage of agar-agar hydrocolloids is the possibility of their repeated use (up to 10 cycles). However, according to physic-mechanical characteristics agar-agar hydrocolloids are significantly inferior to vinyl polysiloxanes. Silicone materials of addition type restore after deformation by 99.5 %, exhibit increased tensile strength, reproduce the relief of details with an accuracy of 2 μm and have unique characteristics of dimensional stability.

REFRACTORY INVESTMENT MATERIALS

Technological process of manufacturing dentures or their parts by pouring or pressing the constructional materials in their liquid state into refractory moulds where they solidify is called ***casting***. In modern dentistry technologies of casting of metal alloys and injection molding of ceramic materials are widely used (fig. 7).

Moulds for casting of metal alloys and pressing of ceramics are made ***of refractory investment materials***. Refractory materials are the materials, which are able to withstand temperature above 1580°C without being destroyed. Refractory properties (heat resistance) are characterized by a temperature at which a standard sample of material in the form of a trihedral truncated pyramid with the height of 30 mm and sides of the bases of 8 and 2 mm (Seiger cone) is softened and deformed with its vertex touching the base.

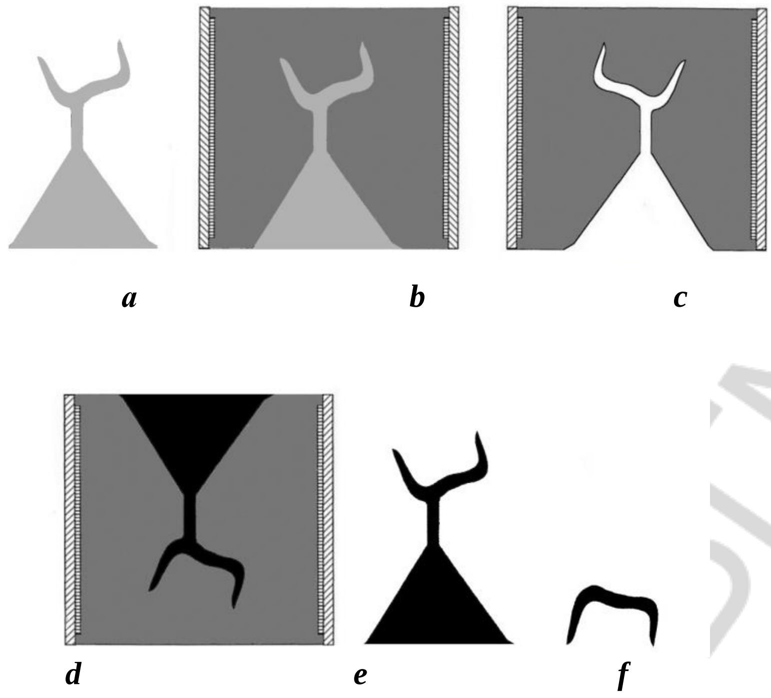

Fig. 7. Sequence of casting of constructional materials: wax model of an artificial crown with the casting (gating) system installed on a sprue base (*a*), production of a mould from refractory material (*b*), mould prepared for casting after melting out of a wax model (*c*), filling of the refractory mould with molten constructional material (*d*), a cast (ingot) of constructional material eliminated from the mould (*e*) and an artificial crown separated from the casting (gating) system (*f*)

There are three types of refractory materials:

− actual refractory materials (heat resistance of 1580–1770 °C);
− highly refractory (1770–2000 °C);
− super-refractory material (above 2000 °C).

Casting refractory mould, as a receiver of the molten metal, should provide the conditions for obtaining high-quality castings. Therefore, investment mixtures should meet certain requirements:

1) should be harmless upon handling;
2) their dispersity has to ensure smooth surface of the casting;
3) dosing of components should be simple and easy to use;
4) mixing time and working time should be 4–6 minutes;
5) refractory mixtures in their liquid phase must have high flowability and ability to wet wax patterns without formation of air pores;
6) hardening time should not exceed 60 minutes;
7) should be strong, and have to ensure integrity of the casting mould during its transportation and pouring;
8) should not disintegrate under the temperature exceeding melting temperature of the constructional material by 200 °C;

9) should possess gas permeability for elimination of vapors and gases formed during heating and casting;

10) should not change chemical composition of the melt and should not react chemically with it;

11) should provide necessary expansion in order to compensate for the metal alloy shrinkage;

12) should not contain substances that can degrade casting quality by reacting with it;

13) should be easily separated from the casting.

A particularly important requirement for investment materials is compensation of metal alloys shrinkage observed during their solidification. Shrinkage of gold alloys during solidification reaches 1.5 %, while shrinkage of chrome-nickel alloy during solidification reaches 2.4 %. Consequently, shrinkage of constructional alloys upon solidification should be compensated by expansion of the mould by the volume of shrinkage. A slight expansion of the mould is already provided during hardening of the investment material. Creation of an expanding mould is mostly promoted by its heating in a muffle furnace.

Refractory materials are mainly produced on the basis of mineral raw materials by formation of a chemical-mineral composition and structure during processing of raw materials. Raw materials used for production of refractory materials include such natural substances as quartzites, quartz sands, refractory clays and kaolins, bauxites, aluminum silicates, hydrated natural varieties of aluminum, magnesites, dolomites, limestones, natural silicates and magnesium hydrosilicates, zircon sands, graphite, and etc.

Industrial production of refractory materials is carried out in the form of dry powdered mixtures of various grinding degrees, which strength characteristics and refractory properties are determined by inclusion of certain binding additives.

Refractory materials used in dentistry for the production of casting moulds are classified by chemical-mineral composition:

Type	**Group**	**Mass percentage of basic chemical components, %**
Silica	Quartz	SiO_2 not less than 98
Aluminous	Corundum	Al_2O_3 more than 95
Zirconium	Oxidzirconium	ZrO_2 more than 85
Carbonaceous	Graphitized	C more than 96

Depending on the designation, the mixtures are divided into facing, filling and unified.

The facing mixture has the highest quality and is used for covering of the working surface of the mould, which is in direct contact with the molten metal.

Thickness of the facing mixture layer depends on the type and nature of casting (15–50 mm).

The filling mixture is poured over the facing mixture, and has lower strength and gas permeability, and is cheaper. The filling mixture is prepared by recycling of previously used investment mixture with addition of 3–5 % of fresh materials.

The unified mixture makes the entire volume of the mould and is widely used in dentistry for casting technologies. It differs from the filling mixture by a large content of fresh materials and better physical and mechanical properties.

Silica (quartz) investment materials are the most widely used investment materials in dentistry. Silicon dioxide, quartz (SiO_2) is a solid, refractory substance. Melting point of quartz is 1800 °C. In nature it is found in the form of transparent crystals, which are called clear (rock) crystals. Depending on the impurities, quartz can have various shades. Quartz crystals (fig. 8, *a*) are subjected to primary calcination — fritting, which results in a product called frit. As a result of rapid cooling of the frit, high stresses are generated inside the melted quartz and lead to extensive cracking of the mass (fig. 8, *b*). The obtained material is easily grinded into a fine powder (fig. 8, *c*).

a ***b*** ***c***

Fig. 8. Quartz crystals (*a*), quartz after fritting (*b*), grinded quartz (*c*)

Under normal conditions, silicon dioxide is most often found in the polymorphic modification of α-quartz, which reversibly transfers into β-quartz at the temperature of 573 °C. With the further increase in temperature, quartz turns into tridymite and cristobalite. Polymorphic transformations of quartz and cristobalite from the α-form to the β-form are accompanied by an increase in volume and are used to compensate for the shrinkage of the casting. The melting point of β-quartz is 1610 °C, and of β-cristobalite — 1723 °C. Tridimite modification of quartz is not used in the manufacturing of refractory moulds since its allotropic transition during temperature increasing is not accompanied by a significant increase in volume.

Refractory investment materials of ***silica type*** contain grinded quartz (marshalite, or powder quartz — fine dispersed mineral filler made from quartz sand containing not less than 98 % SiO_2) and cristobalite (pure fine quartz sand with crystalline structure, processed at high temperatures).

High degree of dispersion of the investment material powder particles ensures good cleanliness of the casting surface. The granularity of the powder from 20 to 60 microns makes up to 90 % of the volume of the granulometric composition of the modern quartz investment materials.

To bind the silica filler particles, sulfate, phosphate and silicate bonds are used. Their names determine subgroups of quartz refractory investment materials (fig. 9).

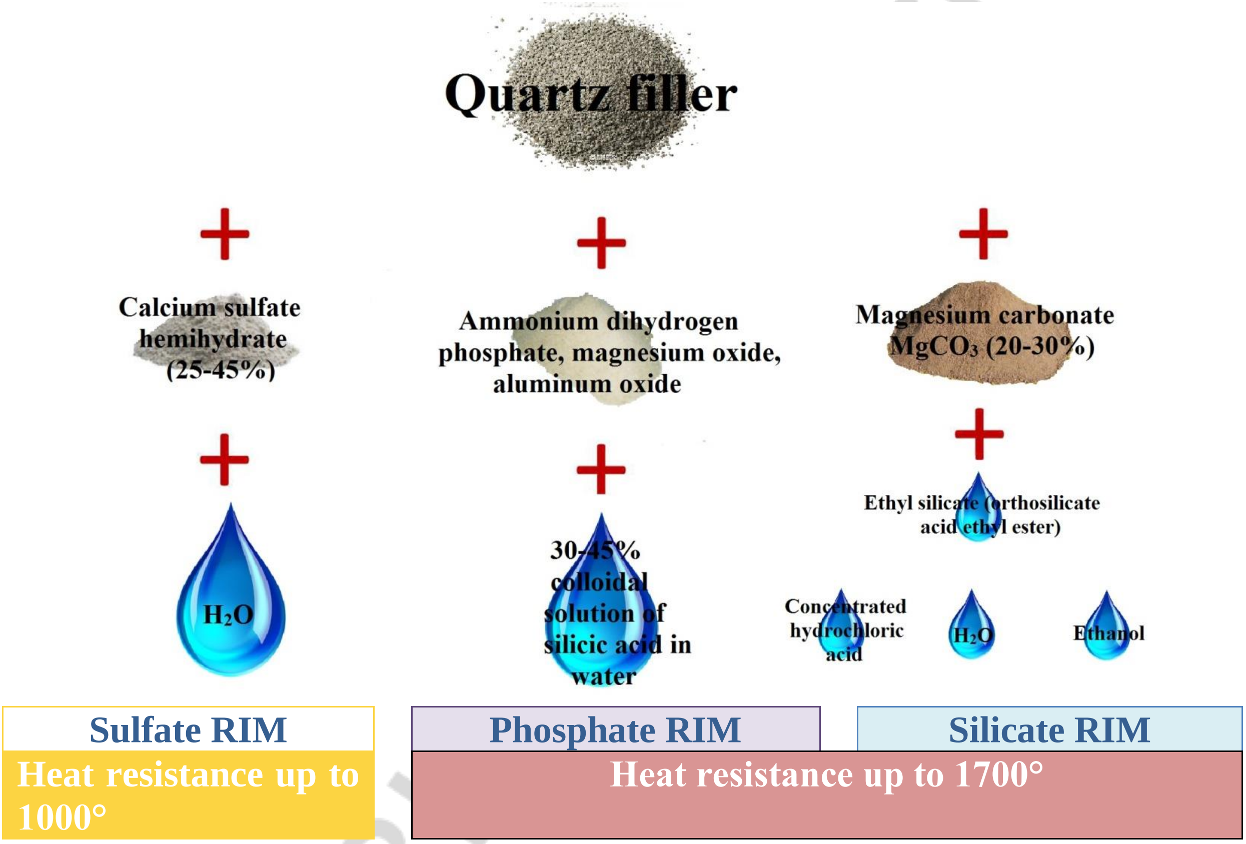

Fig. 9. Compositions of sulfate, phosphate and silicate investment refractory materials

Sulfate quartz refractory investment materials consist of modifications of silica dioxide (filler) and binder — calcium sulfate hemihydrate, which is added to the filler in a volume of 25 to 45 %. After addition of water the investment mixture hardens within 10–30 minutes. During hardening the sulfate investment materials expand within 0.1–0.45 %. In a hard metal ring, in which the investment material is hardening, one should install a seal from a compressible material (dry cellulose or mineral silicate wool with acrylic acid binding agent) in order to allow the expansion proceed in the radial direction during hardening.

Thermal expansion of the mould is the main condition for compensation of the alloys' shrinkage during casting. In order to create a volumetric expansion, before work the mould is exposed to thermal processing. If the molding material contains quartz, the mold is heated up to 700 °C, if cristobalite — up to 450 °C. Thermal expansion rate is determined by the ratio of water and powder during the mixing of the investment material (the thicker the mixture, the greater the thermal expansion of the mould) and the percentage content of cristobalite in the filler (cristobalite causes thermal expansion of the investment material up to 1.6 %, and quartz — up to 1.4 %).

Sulfate investment materials are characterized by low refractoriness not meeting the required criteria. Low indices of «fire resistance» are caused by thermal instability of gypsum. Already at a temperature of 1000 °C gypsum decomposes into calcium oxides and sulfur. Insufficient refractoriness makes it possible to use these investment materials for casting of gold-containing alloys with the melting temperature not higher than 1000 °C.

In ***silicate quartz refractory investment materials***, a hydrolyzed ethyl silicate is used for binding of the silica dioxide modifications. Ethyl silicate is obtained from silicon tetrachloride. As a binding component, ethyl silicate has a crystallographic structure close to the parameters of the crystal structure of the dispersed quartz powder. A hydrolyzed solution of ethylsilicate is prepared by mixing of 10–50 % ethylsilicate, 5–15 % H_2O, up to 1.0 % HCl, and the rest is an organic solvent (ethanol). Preparation of hydrolyzed silicate is carried out in the following sequence. Ethanol, water and hydrochloric acid are poured into the hydrolyser and mixed for 1–2 minutes. Then the mixing is continued, and ethyl silicate is added into the solution.

Silicate investment materials are characterized by high coefficient of thermal expansion and high thermal stability (up to 1700 °C), which allows them to be used for casting of all types of metal alloys used in dentistry, with the exception of titanium.

Phosphate quartz investment materials have fire-resistance of up to 1700 °C. They consist of modifications of silicon dioxide (filler) and a binder — zinc-phosphate cement. When the powder (quartz-cristobalite filler, ammonium dihydrogen phosphate, magnesium oxide, aluminum oxide) is mixed with the liquid (30–45 % colloidal solution of silicic acid in water), phosphates are formed. They firmly bind the particles of the filler of the investment mixture with the phosphate crystallohydrate. The ratio of silica sol to water during the preparation of the investment mass influence the degree of the material expansion. With a decrease in the volume of water, the greater expansion of the investment material is observed. Usually the proportion of three parts of the silica sol liquid per one part of distilled water is used. As a result of thermal processing, phosphates transfer from ortho- to pyroform (pyrophosphate), which has a high refractoriness.

For the casting of titanium (melting point 1660± 20 °C), aluminous high-refractory molding materials with an electrocorundum content of more than 95 % (refractory and chemically resistant material based on aluminum oxide Al_2O_3) are used. Ethylsilicate is used as a binder. Aqueous solutions of magnesium and zirconium salts can be also used as binding materials. In addition to aluminous materials, zirconium refractory materials with ZrO_2 content of more than 85 % bound by zirconium nitrate or silicate and carbonaceous (graphite) investment materials with a resin binder are used as refractory materials for production of casting moulds intended for filling with molten titanium.

TEST QUESTIONS

1. Technological process of products manufacturing by filling of previously prepared forms (moulds) or by plastic deformation of material in accordance with prepared forms is called?

a) formation;
b) investing;
c) refraction.

2. Which of the following investment materials are used for duplication of gypsum models?

a) low fusible metal alloy;
b) agar-agar;
c) alginate;
d) plaster;
e) Vinylpolysiloxanes.

3. Investment materials of which category are used for casting of constructive metal alloys?

a) fusible metal alloys;
b) elastomeric;
c) refractory;
d) thermoplastic.

4. At what temperature is agar gel zolated?

a) 30–10 °C;
b) 10–50 °C;
c) 70–100 °C;
d) 50–60 °C.

5. Technological process of manufacturing dentures or their parts by pouring or pressing the constructional materials in their liquid state into refractory moulds where they solidify is called …

a) investing;
b) modeling;
c) sintering;
d) casting.

6. Refractory materials are the materials, which are …*(multiple choice)?*

a) able to withstand the load over 100 MPa.
b) able to withstand temperature above 1580 °C without being destroyed.
c) used as investment materials for duplication of gypsum models.
d) used in the procedure of casting of metal alloys and pressing of ceramics.

7. What is the role of expansion of the refractory mould by the volume? *(write the correct answer).*

8. In quartz refractory materials quarts plays a role of a … .

a) binder;
b) filler;
c) holder.

9. Which binders are used in quartz refractory materials applied in dentistry?

a) Sulphate;
b) Nitrate;
c) Phosphate;
d) Silicate;
e) Alginate.

10. Which investing materials are used for casting of gold-containing alloys?

a) Silicate quartz RIM;
b) Sulphate quartz RIM;
c) Corundum.

11. Which quartz investment materials have temperature resistance of up to 1700 °C?

a) Phosphate RIM;
b) Sulphate RIM;
c) Silicate RIM.

12. Which refractory materials can not be used for casting of titanium alloys?

a) Sulphate RIM;
b) Silicate RIM;
c) Phosphate RIM;
d) RIM on the base of aluminum oxide.

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