SATURATION OF GLASS PARTICLES WITH METAL DURING SINTERING OF A COMPOSITE MATERIAL OF THE IRON-CAST IRON-GLASS SYSTEM

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Abstract
Sintering of multicomponent systems is characterized by a number of features, consisting in the fact that sintering of dissimilar materials is a complex eutectic process. Along with self-diffusion, which causes the transfer of mass to the contact area of the particles, there is mutual diffusion, which ensures the homogenization of the composition by equalizing the concentrations of unlike atoms within the sample. Under conditions of limited solubility or complete insolubility of the components, sintering of the system is complicated by the isolation of homogeneous particles from mutual contact, hindering self-diffusion and thereby worsening the sintering conditions.

The saturation of particles of vacuum glass brand S88-5 (GOST 11.027.010-75, Russia) and glass «Pyrex» (TS, Russia), which are part of iron and «iron-cast iron glass» materials, depending on the sintering temperature, has been studied.

To improve the interfacial interaction, and, consequently, to increase the mechanical and tribotechnical properties of powdered iron-glass materials, the effect of the glass melting temperature on the saturation of glass with metal was investigated. The dependences of the content of base metal and silicon ions in glass on the sintering temperature are plotted. The effect of the glass melting temperature on increasing the saturation of glass with metal is predicted.

The saturation of the glass with the base metal depends on the viscosity of the glass at sintering temperatures and is accompanied by an increase in microhardness and refractive indices up to 1.2 times compared to the initial state of the glass.

The microhardness of particles of vacuum glass of grade S88-5 after sintering of metal-glass samples increases noticeably than in samples with Pyrex glass.

When sintering «iron-cast iron-glass» materials, the metal base is saturated with silicon, which leads to an increase in the hardness of iron up to 1.8 times.

Keywords: interdiffusion, sintering, pressing, composite material, sitall, glass saturation, microhardness, viscosity.

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1. Introduction
During sintering of metal-glass materials, glass is sitalized [1] and it is necessary to develop such modes of glass that would ensure crystallization in the entire volume of glass inclusions, which contributes to an increase in the strength of the material [2]. In addition, when glass is in contact with iron for a long time in the temperature range 1000–1200 °C, the glass becomes greenish, which indicates its saturation with iron [3].

To improve the mechanical properties and wear resistance of iron of glass materials obtained by cold pressing with subsequent sintering in a protective environment, it is necessary to add components that have better glass wettability to the composition of the charge [4]. The use of cast iron powder as part of the charge leads to such an effect [5]. Since cast iron contains a relatively large amount of silicon and manganese, which, during heating, form corresponding hardly reducible oxides, which improve the wettability of the metal frame with glass during sintering [6].
Saturation of glass is accompanied by an increase in the refractive index [7], and under certain conditions, a crystalline phase may appear in it, which includes ions of iron and other metals [8].

In sintered metal-glass materials, glass particles are surrounded by metal on all sides, which creates favorable conditions for saturation with metal oxides [9]. With an increase in the sintering temperature, the glass becomes more fluid, the contact area between the metal and the glass increases, and, consequently, the possibility of saturating it with metal oxide increases [10].

2. Materials and methods of research

The saturation of particles of vacuum glass grade S88-5 (GOST 11.027.010-75, Russia) and glass «Pyrex» (TS, Russia), which are part of iron and «iron-iron glass» materials, depending on the sintering temperature, has been studied.

The metal basis of the samples used for the study consisted of: powders of iron, gray cast iron obtained by grinding, as well as stearic acid zinc for the perfumery and cosmetic industry. Iron powder grade PZhRV 2.200.28 (GOST 9849-86, Sulin) was used in a fineness of \(-160+50\ \mu m\). Cast iron powder of grade SCH – 20 obtained by mechanical grinding had a fineness of \(-100+50\ \mu m\). The sizes of glass particles were in the range of \(-100+50\ \mu m\), and the amount of glass and cast iron was 6 and 30 \% (by weight), respectively. To improve the compressibility of charge materials, stearic acid zinc for the perfumery and cosmetic industry was used – grade «Ch» (TU 6-09-4473-77, Russia), in an amount of 1.0 \% (by weight) [7].

Samples with dimensions 55×10×10 mm were pressed on a hydraulic press model HPM-100S (Russia) at a constant pressing pressure of 1000 MPa. Glass-metal specimens with a porosity of 8–16 \% were sintered in the temperature range 1000–1200 °C for 1 hour in an endothermic gas environment in a continuous furnace.

The microhardness of glass particles was investigated using a PMT-3 microhardness meter. The weight of 50 g was selected in such a way that glass cracking did not occur when measuring microhardness. On each sample, sintered at a certain temperature, 30 measurements of microhardness were carried out and the percentage composition of the obtained values was determined.

3. Results and discussion

In the process of sintering of powdered iron-glass materials, the bond between the metal base and the glass phase is improved.

By the method of micro-X-ray spectral analysis when comparing the intensities of the excited rays in the samples and standards, the average content of saturated metal and silicon in glass particles that were part of iron-glass, iron-cast iron-glass materials was determined (Fig. 1).

With increasing sintering temperature, the color of glass particles in the samples changed. In the iron-glass with vacuum glass of the S88-5 grade – from colorless to green, and in the iron-glass with Pyrex glass – to black. In the «iron-glass» ones with vacuum glass of the S88-5 brand – up to light green, and in the «iron-glass» ones with «Pyrex» glass – up to violet. The change in the color of the glass particles indicated its saturation with metal ions (Fig. 1).

Fig. 2, 3 show the change in the content of base metal ions and silicon in glass from the sintering temperature. With its increase, the saturation with a metal increases, and a direct relationship is seen between the saturation of glass with a metal and the affinity of this metal for oxygen. The most saturated particles are particles with vacuum glass of grade S88-5 (Fig. 2), the least – with glass «Pyrex», which were part of the «iron-cast iron-glass» samples.

The saturation of glass with metal is significantly affected by the melting temperature of the glass. Thus, in metal-glass samples with Pyrex glass, the saturation of glass particles with metal and a decrease in the silicon content is less intense than in vacuum glass of S88-5 grade. This is due to the fact that Pyrex glass at the same temperatures has a higher viscosity [11] than glass of grade S88-5, due to which the contact between the metal and glass deteriorates, the mobility of the glass decreases. In addition, with an increase in the sintering temperature, the content iron in the matrix decreases and silicon increases.
An increase in the silicon content in the matrix indicates that with an increase in the sintering temperature (especially at 1150–1200 °C), the matrix is saturated with glass components. In turn, the glass is saturated with iron, that is, mutual diffusion occurs between the matrix and the glass phase.

Fig. 1. The average content of saturated metal and silicon in glass particles that were part of iron-, «iron-cast iron-glass» materials: a, b – iron-glass; c, d – «iron-cast iron-glass»

Fig. 2. Changes in the iron content in glass, depending on the sintering temperature of metal-glass samples: 1, 2, 3 – iron cast with vacuum glass of grade S88-5; 4, 5, 6 – iron-cast iron with Pyrex glass

Fig. 3. Changing the silicon content in glass, from the sintering temperature of metal-glass samples: 1, 2, 3 – iron-cast iron with Pyrex glass; 4, 5, 6 – iron-cast iron with vacuum glass, grade S88-5
For the construction of graphic images (Fig. 2, 3), particles of approximately the same size were selected. When the size of the glass particle changes, its saturation with the base metal also changes. For example, in iron-glass samples with Pyrex glass sintered at a temperature of 1150 °C, an increase in the size of a glass particle by 1.5 times led to a drop in the iron content from 9 to 5%.

Usually towards the edge of the glass particle, the saturation metal content increases. In addition, even at temperatures of 1000–1050 °C, crystals of precipitating metals are noticeable in the glass. Consequently, the glass is no longer homogeneous in chemical composition.

Fig. 4 shows the determination of the refractive indices of glass particles that were part of the sintered samples. At a temperature of 1050 °C in iron-glass specimens with glass of grade S88-5, the refractive index ranges from 1.514 to 1.523; at 1150 °C from 1.521 to 1.537; at 1200 °C from 1.529 to 1.580. Apparently, glass with a low refractive index corresponds to those areas of the particle where minerals are released. In general, an increase in the sintering temperature of metal-glass samples with S88–5 glass leads to an increase in refractive indices (Fig. 4).

![Fig. 4. Change in the refractive index of vacuum glass of grade C88-5 from the sintering temperature of metal-glass materials: material: 1 – iron-glass, 2 – «iron-cast iron-glass»](image)

In samples with Pyrex glass (Fig. 5), an increase in the refractive index is observed in iron and iron-cast iron-glass samples up to a certain limit (1150 °C), then the refractive index of the glass decreases. The drop in the refractive index in the samples sintered at temperatures above 1150 °C is obviously associated with the intense crystallization of the glass. In iron-glass samples sintered at a temperature of 1250 °C, it was not possible to measure the refractive index, since non-crystallized glass could not be separated from the sample.

Most of the minerals released in glass during sintering of metal-glass minerals have a higher hardness than glass. For example, the microhardness of quartz is 10230–12360 MPa; magnetite – 5350–6950 MPa; fayalite Fe₂SiO₄ – 6890–7480 MPa, etc. [12]. Consequently, the release of minerals should affect the microhardness of the particles that make up the sintered metal-glass materials. Table 1 shows the limits of the microhardness of the glass phase of the samples sintered at temperatures from 1000 to 1200 °C.

From the Table 1 it is possible to see that with an increase in the sintering temperature, the spread of microhardness values increases and in all samples a tendency to an increase in microhardness is observed. The microhardness of the glass phase of samples with S88-5 glass increases especially. In iron-glass specimens sintered at 1200 °C, 17% of all measurements fall on areas
with a microhardness of 10780 MPa and 29% on areas of microhardness (7430 MPa) are almost twice as high as that of the original glass.

![Graph showing refractive index change](image)

**Fig. 5.** Change in the refractive index of Pyrex glass from the sintering temperature of metal-glass materials: material: 1 – iron-glass, 2 – «iron-cast iron-glass»

| No. | Sintering temperature, °С | Microhardness of the glass phase in materials, MPa |
|-----|---------------------------|--------------------------------------------------|
|     |                           | with glass of C88–5 brand                       |
|     |                           | iron-glass | iron-cast iron-glass | iron-glass | iron-cast iron-glass |
|     |                           | from       | to                     | from       | to                     |
| 1   | 1000                      | 4280       | 6360                   | 3890       | 5776                   | 3420       | 7030                   | 3480       | 5200                   |
| 2   | 1050                      | 4980       | 7430                   | 4540       | 6360                   | 4800       | 6380                   | 3400       | 5000                   |
| 3   | 1150                      | 3800       | 8400                   | 4900       | 8800                   | 4240       | 7100                   | 3450       | 4950                   |
| 4   | 1200                      | 4874       | 10780                  | 4876       | 8910                   | 3850       | 7200                   | 3450       | 5330                   |

Table 1

Limits of microhardness of the glass phase of the samples

In specimens with Pyrex glass, the spread of microhardness values is small, especially for «iron-cast iron-glass» specimens, and it does not increase as noticeably as in specimens with S88-5 glass. This is explained by the fact that metal-glass samples with Pyrex glass are less crystallized, as established by metallographic analysis. The reason for this is that Pyrex glass at the same temperatures has a higher viscosity [11] than with C88 glass-5. Due to this, the contact between the metal and glass worsens, and the mobility of the glass decreases. As a result, the matrix is not completely wetted with the Pyrex glass.

Intensive crystallization of glass at temperatures of 1150–1200 °C in samples with glass of the S88–5 brand is accompanied by a sharp increase in the average microhardness (Fig. 6). In samples with Pyrex glass, its crystallization and saturation with metal is less intense, which affects the microhardness. As can be seen from Table 1, in the temperature range 1000–1050 °C, a decrease in microhardness is observed, especially its lower limit. In samples with S88-5 grade glass, such a drop is not observed. This is due to the fact that, at these temperatures, the Pyrex glass has a significantly higher viscosity than the C88–5 glass and the gas released when the samples are heated, and great obstacles are created for reaching the surface. Due to the fact that particles of Pyrex glass at these temperatures contain many gas bubbles, this leads to a decrease in microhardness.
**Fig. 6.** Change of microhardness of glass particles of metal-glass materials from sintering temperature. With vacuum glass of grade C88-5: 1 – iron-cast iron; 2 – iron with Pyrex glass; 3 – iron-cast iron; 4 – iron

By measuring the microhardness of «iron-cast iron-glass» materials, let’s found that in the samples sintered at temperatures above 1000 °C, the glass becomes very fragile and porous, therefore, when the microhardness changes, the glass regions are destroyed. However, at 1250 °C, there are quite strong glass areas, the microhardness of which is 10157 MPa (**Fig. 7, d**).

**Fig. 7.** Distribution of microhardness in glass particles. a, b – iron with S88-5 grade glass and with Pyrex glass, sintering temperature 1150 °C, respectively; c, d – iron cast with glass of grade S88-5, sintering temperature 1000 and 1200 °C, respectively

At a temperature of 1000 °C, the microhardness of glass particles is in the range from 4280 to 5200 MPa, then it drops sharply. At 1150 °C – 1200 °C, along with brittle areas, there is a small number of areas with high microhardness. At a temperature of 1000 °C, apparently, saturation of the metal base with silicon occurs, since the hardness of iron at the boundary with glass increases to 4280–4980 MPa, although further from the boundary the microhardness drops to 1200 MPa (**Fig. 7, c**). At a temperature of 12000 C, the microhardness of the iron base metal was 5200–5330 MPa. In some cases, when the sizes of crystalline inclusions allowed for detailed measurements, the microhardness of some minerals was determined. As a rule, the microhardness of the crystalline phase was higher than that of glass (**Fig. 7, a, b**). For example, the microhardness of crystals of the «iron-cast iron-glass» base in the «iron-cast iron-glass» samples was equal to 9780–10487 MPa.

The results obtained and the recommendations given can be applied in the ranges of conditions that apply to parts of household appliances operating under conditions of intense wear at
a contact load of 2–8 MPa, friction rate $V = 2.1$ m/s. The highest mechanical and antifriction properties are exhibited by samples containing 30–50 wt % cast iron with particle sizes from 100 to 160 μm and 2–6 wt % glass powder (50–200 μm), the rest being iron.

The results obtained substantiate the use of the investigated powdered iron-cast iron-glass composite material with a porosity of 8–16 % as an antifrictional purpose for parts of household appliances.

4. Conclusions

The proposed approach to the problem of saturation of glass with a metal, taking into account mutual diffusion, makes it possible to establish: saturation of glass with a base metal depends on the viscosity of glass at sintering temperatures and is accompanied by an increase in microhardness and refractive indices; the saturation of glass with the base metal depends on the degree of affinity of this metal for oxygen.

It is shown that the microhardness of particles of vacuum glass of grade S88-5 after sintering metal-glass samples increases by 27 % than in samples with Pyrex glass.

When sintering «iron-cast iron-glass» materials, the metal base is saturated with silicon, which leads to an increase in the hardness of iron by 1.8 times.

The results obtained substantiate the use of the investigated powdered iron-cast iron-glass composite material as an antifriction material.

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