# EXPANDED GLASS BY A NONCONVENTIONAL MICROWAVE HEATING TECHNIQUE FROM RESIDUAL GLASS

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ABSTRACT: Glass-ceramic foam was produced from residual glass (87.6-87.9 %), coal fly ash (8.5-10 %), silicon carbide (2-3 %), and kaolin (0.4-0.6 %) by sintering at 950-975 ºC. The heating technique was original, being used the preponderantly direct warming procedure using electromagnetic waves. The glass-ceramic foam had the following characteristics: apparent specific gravity between 0.22-0.28 g/cm$^3$, porousness between 86.7-89.5 %, thermal conductance in the range 0.055-0.065 W/m·K, compression resistance between 1.8-2.5 MPa, water absorption below 1.2 vol. %, and pore dimension under 1 mm. The specific electricity consumption had very low values (between 0.75-0.83 kWh/kg). The application field of the product includes thermal insulation materials for construction, architectural components and other applications that do not require resistance to high mechanical stress.
KEYWORDS: coal fly ash, compression resistance, expanded glass, residual glass, microwave warming, silicon carbide.

## 1. INTRODUCTION

The large amounts of waste (plastics, metals, glasses, textiles, paper, and cardboard) generated by consumption and the end-of-life of many utilities of everyday life that have invaded the human civilization by throwing them in landfills seriously affect the health human and of the planet. The concerns of the modern world for mitigating these effects through the recycling of waste and its use as raw materials, especially in industrial production, began to manifest themselves in the 1970s with the onset of the oil crisis, which affected humanity in terms of energy. The need to replace energy-intensive technological processes with the much lower-use consumption of recycled products in form of waste had a double effect: disposal of some waste and energy saving. At the end of the last millennium, the danger of destroying the planet's ozone layer due to greenhouse gas emissions also played an important role in the waste recycling process, so that today, the problem of reducing the danger of landfills by recycling them has gained a global character.

The glass is a waste whose generation rate is constantly increasing. The sources of generating this waste are mainly post-consumer container glass and shards of glass resulting from demolishing and rehabilitation of the civil constructions. Of course, the industrial production of newly created glass uses residual glass as raw material, but the necessary process of selecting coloured glass is expensive. Since the middle of the 20$^{th}$ century, it has been discovered that the glass foam leads to light porous products, sufficiently mechanically resistant, resistant to fire, moisture, corrosion, and with a very high durability, usable as substitutes for some construction materials. Pure glass was used in the beginning. Since the 1980s, the first glass foam plants appeared, using recycled residual glass.. In recent decades, this activity has developed and extended in more European states (Belgium, Switzerland, Austria, Germany, Scandinavian states, etc.), the US, and China. Manufacturing prescriptions used in industrial production are focused on common types of residual glass from the soda-lime-silica glass category and pore-making agents, used to expand the raw material based on glass heated to temperatures corresponding to its softening point, are materials of whose effect is well known in the foaming process (coal, black carbon, glycerol, silicon carbide, calcium carbonate) [1]. Also, the tunnel type-warming furnaces with conveyor belt, use exclusively conventional warming methods (electric resistances or fossil fuel consumption).

Recently, numerous small-scale experiments have been performed by research teams around the world, extending the field of residual glass (cathode ray tube glass, borosilicate glass, residual glass mixed with various silicate wastes such as coal ash, incinerator ash, oil-shale, metallurgical slag, zinc-hydrometallurgy waste, red mud, etc.) [2-9] or pore-making agents (carbonates, nitrates or natural products with a high calcium carbonate content such as egg shell, clam shell, etc.) [2, 10-15].

The improving trend of the manufacturing processes of glass foams from recycled residual glass has also manifested itself in the heating method of the raw material. Recently, the Romanian company Daily Sourcing & Research has performed several and various tests in which the microwave irradiation of the glass powder has been applied exclusively. The adopted technical solution, characterized by preponderantly direct warming procedure using electromagnetic waves and partially indirect warming by conventional heat radiation, proved to be optimal allowing reaching a significantly higher warming rate than that normally used by conventional methods, the homogeneous microstructural configuration of the expanded material not being affected. On the other hand, the specific electricity consumption has been greatly reduced, even in the unfavourable conditions of small-scale work.

The current paper refers to a foaming method of residual glass by using a by-product of thermal power plants (coal fly ash) and silicon carbide (SiC) as a pore-making agent. The results obtained in experimental conditions both by conventional heating [16] and by nonconventional microwave heating performed in the Romanian company [17] are known. According to the paper [16], the raw material mixture included 80 % residual glass and 20 % coal fly ash, the addition of SiC as a pore-making agent being reduced to 2 %. The optimum heating temperature was experimentally determined between 1000-1050 ºC, at which the material porosity was 75 % and the highest homogeneity of the pore network was obtained. The apparent specific gravity of the expanded material had values within the limits 0.2-0.4 g/cm$^3$ and the compression resistance at the optimal process parameters had the value of 1.5 MPa. The experiment presented in [17] reduced the coal ash proportion in the starting mixture to values between 3-11 % and tested SiC proportions between 2-5 %. The optimum composition of the mixture contained 87.5 % residual glass, 9.5 % coal fly ash, 3 % SiC, and 10 % additional water (as a binder). The optimum sinterization temperature decreased to 980 ºC, unlike the values achieved in [16]. The product characteristics in the best variant were: the apparent specific gravity of 0.24 g/cm$^3$, porousness of 88 %, thermal conductance of 0.052 W/m·K, compression resistance of 1.25 MPa, and pore dimension within the limits 0.4-1.2 mm in a homogeneous microstructure.

The addition to the starting mixture of silicate waste such as those mentioned above contributes to the formation of the so-called "glass-ceramic", which are produced by a controlled crystallization (devitrification) of the glass [4]. Glass-ceramics are polycrystalline materials that can contain 50-90 % crystalline phases, the rest of the composition being characterized by a non-crystalline (amorphous) phase. The glass-ceramic foam is adequate as materials for buildings and constructions that require insulation properties combined with mechanical resistance.

The objective of this paper was increasing the compression resistance of the expanded product by adding low proportions of kaolin, under conditions of keeping in principle the composition of the starting mixture and using the same own original technique of preponderantly direct warming procedure using electromagnetic waves (microwaves).

## 2. METHODS AND MATERIALS

### 2.1 Methods

The main method industrially used for expanding the residual glass in form of fine powder is the incorporation of an expanding agent which has the ability to release a gas or a gaseous compound through a chemical reaction that takes place at a high temperature corresponding to beginning the viscosity decrease of the glass mas. The concordance between the value of this temperature point and the temperature range in which the gas releasing reaction takes place is important for the released gas to form bubbles and to remain blocked in the molten glass [1].

SiC is one of the most efficient foaming agents in the glass expansion process, being together with coal and calcium carbonate the most commonly used pore-making agent in the industrial cellular glass manufacturing [1].

In accordance to [18], SiC oxidizes to above 900 ºC by two possible reactions, resulting $CO_2$ and/or CO, which contribute to glass foaming as well as $SiO_2$ and/or SiO, that enter into the molten glass composition.

$$SiC + 2O_2 = SiO_2 + CO_2 \quad (1)$$

$$SiC + O_2 = SiO + CO \quad (2)$$

Coal fly ash is usually the alumino-silicate material added to the glass-based raw material when SiC is used as an expanding agent. This is a by-product resulting from the process of coal burning in thermal power plant boilers and is captured in electric filters. Coal fly ash contributes to obtaining a foamed product with a good structural homogeneity, its

contribution in the expanding process being the main reason for the use together with SiC in different known experiments from the literature. It has been found that high weight proportions of coal ash in the starting material mixture lead to increasing the thermal level of the sintering and expanding the glass and therefore, to increasing the energy consumption. This is one of the reasons why industrial glass expanding prescriptions do not usually include the coal ash.

Previous experiments performed with mixtures including residual glass, coal fly ash, and SiC led to manufacturing products whose compressive strength had acceptable values for the field of application (thermal insulation materials for building), but quite low (below 1.5 MPa). The method proposed in the present work is the addition of low weight ratios of kaolin powder. Its use in foaming processes helps to increase the mechanical abilities of kaolin-based products [18]. In the industrial making of cellular glass gravel is reported the use of small amounts of this material in the manufacturing prescriptions of Glapor Werk Mitterteich Company [20].

According to [21], kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) is a clay mineral. Commercially, it is found as a dry powder, semi-dry noodle or liquid slurry. By heating in air at atmospheric pressure, i.e. in the conditions in which the experiment takes place, kaolin undergoes several phase transformations. Its dehydration begins at 550-600 ºC producing metakaolin, but the hydroxyl loss continues up to 900 ºC. The heating at 925-950 ºC leads to the transformation of metakaolin into an aluminum-silicon spinel ($Si_3Al_4O_{12}$).

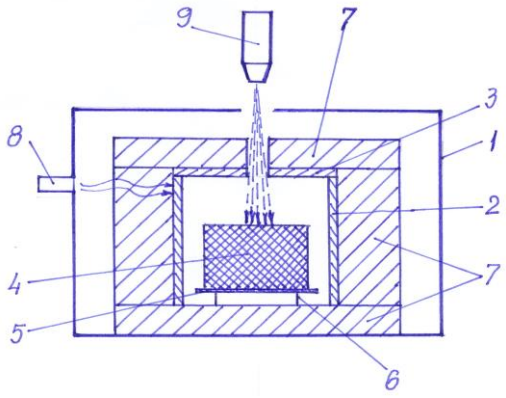
The microwave equipment built for experiments conducted in the last years in the Romanian company Daily Sourcing & Research is based on an electromagnetic wave device commonly used in the household constructively prepared for very high temperature operations (up to 1200 ºC). The oven has a single magnetron (microwave generator) with an installed power of 800 W. Due to limited available inner volume, the rotation mechanism of the support at the oven base has been eliminated. This change did not affect the uniformity of the microwave heating due to the high enough occupancy of the oven enclosure, which does not allow creating areas not covered by the microwave field during the heating process. The mixture for warming was deposited freely in the form of a cylindrical pressed powder on a stainless steel plate placed about 20 mm above the bed of ceramic fiber mattresses bed which ensures the thermal protection of the bottom of the oven. The outer surface of the material was protected with ceramic fiber mattresses. It is important to note that the direct microwave warming is fully different from the conventional warming. The warming processing is beginning in the middle of material, the electromagnetic wave power being turned into heat. Thus, the hottest zone of the material becomes its central area and the heat transfer takes place from the inside to the peripheral material zone. This volumetric heating mode is specific to the direct microwave heating [22]. For this reason, the thermal protection inside the oven aims to protect the irradiated material that concentrates all the captured heat. The walls and vault of the oven no longer need to be protected because the ceramic fiber provides a very advanced insulation, so that at a material temperature of about 1000 ºC, the temperature measured on the unprotected metal walls of the oven does not exceed 60 ºC. Another peculiarity of the direct microwave heating is selectivity [23], i.e. only the target specimen is warmed, not also the other oven components.

The first experimental tests showed that the direct contact of the microwave field emitted by the 800 W-magnetron at 2.45 GHz with a mixture of pressed powder glass also containing a small proportion of pore-making agent causes altering the inner zone of the material structure at the temperature at which its expansion takes place. The solution adopted by the research team was to protect the material with a screen made of 80/20 mixing between silicon carbide and silicon nitride with a thin wall (2.5 mm). The shape of the screen was a crucible or cylindrical tube made in China including a ceramic lid with the same composition. This screen type has the ability to allow predominant penetration of microwaves and, simultaneously, partial waves absorption in the screen wall. The microwave field that crosses the wall achieves a typical direct warming by irradiation, but with a lower intensity that does not cause disturbance to the structural network of glass, while the microwaves absorbed in the wall achieve a rapid and intense warming, the heat being transmitted by thermal radiation to the material.

For controlling the upper surface temperature of the irradiated material, Pyrovar type-radiation pyrometer was positioned above the oven at 400 mm visualizing the hot material through 30 mm-holes provided in the oven upper wall and the ceramic cover on a common vertical axis.

Overall image of the experimental device (a) existing in the Romanian company mentioned above and the scheme of this device (b) are shown in Figure 1.

(a)




(b)

**Figure 1.** Experimental microwave device
(a) – overall image of the experimental device; (b) – scheme of the device: 1 – microwave oven; 2 – ceramic tube; 3 – ceramic cover; 4 – pressed mixture; 5 – stainless steel plate; 6 – metal support; 7 – heat insulation; 8 – waveguide; 9 – pyrometer.

## 2.2 Materials

Raw material adopted in the experiment was composed of residual glass including post-consumer container glass (colourless, green and brown, in approximately equal proportions) and fly ash from the Romanian thermal power plant Paroseni. The oxide composition of the three glass types is indicated in Table 1. The recycled residual glass was cleaned by washing, broken in a crusher, ground in a ball mill, and sorted by sieving, the grain dimension under 80 μm being allowed. The residual glass preparation operations were carried out in Bilmetal Industries SRL Popesti-Leordeni, Romania.

The coal fly ash initially had a grain dimension under 250 μm and had to be ground in a ball mill. The ash grain dimension suitable for use in the experiment was under 100 μm. The oxide composition of the coal fly ash is also shown in Table 1.

**Table 1.** Oxide composition of raw material

| Oxide composition | Colourless glass (wt. %) | Green glass (wt. %) | Brown glass (wt. %) | Coal fly ash (wt. %) |
|---|---|---|---|---|
| $SiO_2$ | 71.7 | 71.8 | 71.1 | 46.5 |
| $Al_2O_3$ | 1.9 | 1.9 | 2.0 | 23.7 |
| CaO | 12.0 | 11.8 | 12.1 | 7.9 |
| MgO | 1.0 | 1.2 | 1.1 | 3.2 |
| $Na_2O$ | 13.3 | 13.1 | 13.3 | 6.0 |
| $K_2O$ | - | 0.1 | 0.1 | 4.1 |
| $Fe_2O_3$ | - | - | 0.2 | 8.6 |
| $Cr_2O_3$ | 0.05 | 0.09 | - | - |
| $SO_3$ | - | - | 0.05 | - |

SiC was used as a pore-making agent. Bought from the trade, the material had the grain dimension under 10 μm and was incorporated into the starting mixture without additional processing.

Kaolin was commercially bought as a dry fine powder. It was also mixed together with the other materials noted above.

In order to promote the cold pressing of the mixture in a metal mould with detachable wall, it was wetted with distilled water as a binder, added to the vessel in which the dry components were mixed. After adding water, a new mixing was performed, followed by axial pressing at a maximum of 5 MPa. The pressed material was removed from the mould as a sufficiently compact body, being deposited freely in the microwave oven.

Three experimental variants were adopted for manufacturing glass-ceramic foam by the nonconventional procedure of preponderantly direct microwave warming, using the own results obtained in the work [14] and adding proportions of kaolin between 0.4-0.6 wt. %. Table 2 presents the composition of the four tested variants.

**Table 2.** Experimental variants composition

| Variant | Residual glass (wt. %) | Coal ash (wt. %) | Kaolin powder (wt. %) | SiC (wt. %) | Water addition (wt. %) |
|---|---|---|---|---|---|
| 1 | 87.6 | 10.0 | 0.4 | 2.0 | 10.0 |
| 2 | 87.7 | 9.5 | 0.4 | 2.4 | 10.0 |
| 3 | 87.9 | 8.5 | 0.6 | 3.0 | 10.0 |

## 2.3 Characterization methods of glass-ceramic foam specimens

Experimental establishing the glass-ceramic foam features was achieved with well-known methods. The apparent specific gravity was determined by the gravimetric procedure [24] and the porousness was established by the comparing technique of the true and apparent density [25]. The compression resistance was measured by the use of TA.XTplus

Texture Analyzer, while the thermal conductance was determined by the heat-flow procedure (ASTM E1225-04). The absorption of moisture was established by the classic method of water immersion (ASTM D570). The specimens microstructure was analyzed with ASONA 100X Zoom Smartphone Digital Microscope. For identification the crystalline phases of the cellular material specimens, X-ray diffractometer Bruker-AXS D8 Advance with CuKα radiation was used according to EN 13925-2:2003.

## 3. RESULTS AND DISCUSSION

### 3.1 Results

As mentioned above, the glass-ceramic foam manufacturing process took place on the 800 W-microwave oven in Daily Sourcing & Research SRL adapted constructively for high temperature operation. The dry raw material had 500 g in all tested variants and the addition of water as a binder was 10% reaching a wet total of 550 g.

Operational data of the procedure including the temperature for each variant, warming duration, warming and cooling speed, index of increasing by expansion of the initial material volume, quantity of glass-ceramic foam, and specific electricity consumption are presented in Table 3.

**Table 3.** Operational data

| Operational date | Variant | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Dry raw material/glass-ceramic foam quantity (g) | 500/ 489 | 500/ 488 | 500/ 490 |
| Process temperature (ºC) | 975 | 970 | 950 |
| Process duration (min) | 39 | 38 | 35.5 |
| Speed (ºC/min) - heating - cooling | 24.5 4.5 | 25.0 4.3 | 26.3 4.3 |
| Index of volume increasing | 1.50 | 1.70 | 2.20 |
| Specific electricity consumption (kWh/kg) | 0.83 | 0.81 | 0.75 |

The examination of operational data in Table 3 allowed observing the influence of the coal fly ash content of the initial mixture on the final process temperature. Thus, for 10 % coal fly ash, the required temperature reached 975 ºC, while for 8.5 %, the temperature value decreased to 950 ºC. Accordingly, the warming duration was also reduced from 39 to 35.5 min, provided that the average warming speed was between 24.5-26.2 ºC/min. Compared to works [13] and [14], the value of the warming rate is comparable. In the current work, a slightly lower average cooling rate of 4.3-4.5 ºC/min was adopted, obtained by extending the storage time in the off oven, with ceramic fiber protection. What is remarkable about microwave warming is the high level of efficiency in terms of energy, the specific electricity consumption decreasing to 0.75 kWh/kg, which coincides with the minimum value reported in the glass foam industry. Given the validity of the assumption made in [26], the heat efficiency of an industrial microwave oven would increase by up to 25 % compared to that of a similar low power oven.

The glass-ceramic foam specimens corresponding to the three tested experimental variants are presented in Figure 2 in cross section. Their appearance indicates good macrostructural homogeneity especially in the case of specimen (c) obtained in variant 3.

(a) (b) (c)

**Figure 2.** Cross section glass-ceramic foam specimens (a) – variant 1; (b) – variant 2; (c) – variant 3.

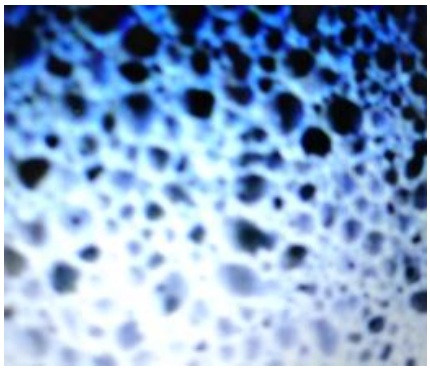
The main characteristics of the cellular glass-ceramic specimens are indicated in Table 4.

**Table 4.** Characteristics of cellular glass-ceramic specimens

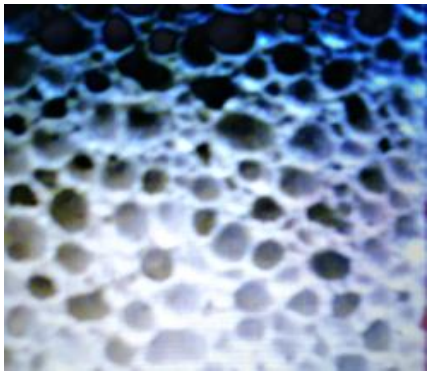
| Variant | Apparent specific gravity ($g/cm^3$) | Porousness (%) | Thermal conductance (W/m·K) | Compression resistance (MPa) | Absorption of moisture (vol. %) | Pore dimension (mm) |
|---|---|---|---|---|---|---|
| 1 | 0.28 | 87.6 | 0.065 | 1.8 | 1.0 | 0.10-0.40 |
| 2 | 0.25 | 88.1 | 0.060 | 1.8 | 1.2 | 0.30-0.50 |
| 3 | 0.22 | 89.5 | 0.055 | 2.5 | 0.9 | 0.40-1.00 |

According to the data in Table 4, all specimens made in this experiment have excellent heat insulation properties. Thus, the apparent specific gravity has low values within the limits 0.22-0.28 g/cm$^3$, the lowest value corresponding to variant 3 and the highest, to variant 1. The specimens porousness varies in high limits (between 86.7-89.5 %) and the thermal conductance registers low values (0.055-0.065 W/m·K) indicating the possibility of achieving very good heat insulation. According to the literature, decreasing the thermal conductance is the effect of kaolin addition. Also, increasing the compression resistance of the material, that reaches 2.5 MPa in variant 3, is due to the addition of kaolin. From the data in Table 4, the moisture absorption is very low (below 1.2 vol. %).

The microstructural appearance of the glass-ceramic specimens experimentally made by preponderantly direct microwave irradiation technique is shown in Figure 3. The examination of the three pictures led to the conclusion that the distribution of pores in the material section is evenly, the microstructural homogeneity characterizing all three materials. The pore dimensions were determined, their values being presented in Table 4.



(a)



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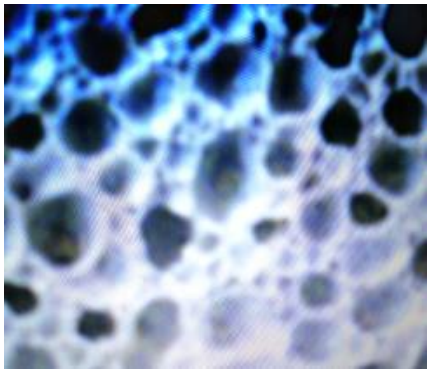

(c) **_____ 1 mm**

**Figure 3.** Microstructural appearance of the glass-ceramic specimens
(a) – variant 1; (b) – variant 2; (c) – variant 3.

XRD analysis for identification the crystalline phases indicated wollastonite ($CaSiO_3$) as the main crystalline phase and quartz, mullite, and silicon carbide (SiC) to a small extent.

## 3.2 Discussion

Making glass foams from recycled residual glass is a process with very large objectives. In 3-4 decades of global activity there has been a great diversification of glass foam types depending on their fields of application. Industrial-scale manufacturing companies have focused on high-demand domains and the adopted manufacturing techniques aim at low energy consumption, easy-processing residual glass from the soda-lime-silica glass category and cheap materials as pore-making agents and other additives.

Although the scientific research in the glass foam making domain is very active worldwide, offering valuable solutions tested in the laboratory or under pilot conditions, the industrial companies do not show interest in technological innovations.

The production of cellular glass from residual glass, fly ash, and SiC as a pore-making agent is a well-known facility for obtaining porous products for thermal insulation materials in construction, but also for architectural components and other applications that do not require mechanical stress. The use of coal fly ash is unattractive for the main manufacturers for economic reasons, because the ash increases the process temperature and thus the energy consumption increases also.

The current paper represents a relatively minor technological improvement of the glass-ceramic foam manufacturing method, aiming to increase by about 66 % the compression resistance of the expanded material by adding kaolin to the starting mixture. The strength increase seems very high, but

in absolute value it means its improvement from 1.5 to 2.5 MPa. The original character of the making process used by the authors is the application of the own preponderantly direct microwave warming technique, currently used on an experimental scale, but which has proven excellent efficiency in terms of energy compared to conventional warming methods commonly used in the glass foam industry.

## 4. CONCLUSION

The work aimed to increase the compression resistance of cellular glass-ceramic made from recycled residual glass, coal fly ash, and SiC by adding a small quantity of kaolin to the starting material mixture. The experiment kept the original own technique of preponderantly direct electromagnetic wave warming, unlike the classic conventional warming methods used in industrial glass foam manufacturing processes. Three experimental variants were tested on the 800 W-microwave oven in the Romanian company Daily Sourcing & Research. The sintering process temperature was in the range 950-975 ºC. The characteristics of the expanded product were: apparent specific gravity between 0.22-0.28 g/cm$^3$, porousness within the limits 86.7-89.5 %, thermal conductance in the range 0.055-0.065 W/m·K, compression resistance within the limits 1.8-2.5 MPa, moisture absorption under 1.2 vol. %, and pore dimension under 1 mm. The specific electricity consumption had very low values (within the limits 0.75-0.83 kWh/kg). The objective of the work was achieved, the maximum compression resistance increasing from 1.5 to 2.5 MPa. The application field of the product includes heat insulation materials for buildings, architectural components and other applications that do not require high-strength to mechanical stress.

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