Study of the effect of shock-wave treatment on the structure and properties of Phenylone

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SUMMARY

The results are given of an investigation of the effect of explosive pressing on the structure and properties of Phenylone S3. The optimum pressure of explosive pressing and the optimum temperature of subsequent sintering of preforms of 3–10 mm thickness were determined. The thermomechanical properties and the structural changes of Phenylone were investigated after shock-wave and heat treatments. The physicomechanical properties of Phenylone S3 after explosive pressing are compared with those of Phenylone S2 produced by hot pressing.

Phenylone (a trade name used in Russia for the linear aromatic \([-\text{NHC}_6\text{H}_4\text{NHOCC}_6\text{H}_4\text{CO}\])_n polyamide poly-meta-phenylene isophthalamide) possesses a fairly high prolonged temperature service, high hardness, rigidity, and strength, and good antifriction and other properties. On account of this, Phenylone is used mainly for the manufacture of components and assemblies undergoing severe service conditions.

There is a variety of Phenylone moulding powders: P, S1, S2, S3, S4. An analysis of studies [1, 6, 7] of Phenylone showed that it is chiefly P, S1, and S2 that have been investigated. There have been practically no studies of Phenylone S3 because it is more difficult to process. Phenylone S3 is a thermoplastic aromatic polyamide with a high heat resistance and a service temperature of up to 270°C. Owing to the increased rigidity of its macromolecule chains and strong intermolecular interaction, this polymer is characterised by low deformability in the region of the softening point and flow temperature, which makes it a difficult material to process by normal methods [1]. A promising method for processing Phenylone is explosive pressing (EP), enabling practically any pressures to be realised and not requiring high-power press equipment [2, 4, 5].

The aim of the present work was to establish the laws governing the effect of explosive pressing and subsequent sintering on the structure and properties of Phenylone S3.

The effectiveness of explosive technologies depends largely on the development stage of the explosive loading schemes. At present, for the pressing of powders, schemes of plane sliding loading in an ampoule are also widely used. The use of a particular explosive loading scheme is dictated by the shape, size, and properties of the preforms produced. In this work, explosive pressing was carried out by a plane sliding loading scheme. The explosive loading parameters are determined by the type and height of the explosive charge. Explosive blends in different ratios were used as the explosive, which ensured a variation in the detonation rate from 1680 to 3400 m/s. Here, according to calculation of the loading parameters by the developed computer program [5], the shock-wave pressure was 0.67–3.0 GPa. Phenylone S3 was used as finely dispersed powder with a bulk density of 0.3–0.4 Mg/m³.

To study the combined effect of explosive and subsequent heat treatment on the structure and properties of Phenylone, different methods of investigation were used. Density was measured by hydrostatic weighing on an ADV-200M analytical balance. Microhardness was measured on a PMT-3 hardness meter under a 0.2 N load.
Impact strength was determined on a pendulum impact device of rigid design on unnotched testpieces (GOST 4647–80). Thermomechanical tests were conducted on a TMI-1 unit under a load of 1 N. Microstructural investigations were conducted by optical microscopy on an Olympus 61BX microscope in reflected light at 200× magnification. The characteristic temperatures of the polymers were studied by differential thermal analysis (DTA) and thermogravimetric analysis (TGA) using a Paulik-Paulik-Erdei derivatograph.

One of the most important indices characterising the process of explosive pressing and the quality of the preforms obtained is their density. It is known [3] that, to prevent excessive heating up of the polymer powder because of its high porosity, and to improve the quality of pressing, before explosive pressing it is necessary to compact the powder to a density of 0.8 Mg/m³. The influence of the explosive detonation rate and the associated pressure of the explosion product and impact momentum on the density of Phenylone showed (Figure 1) that the pressed material has practically the theoretical density (1.32–1.33 Mg/m³) after explosive loading with a pressure of 0.5–1.5 GPa. Increase in pressure to 3 GPa leads to a reduction in the density of the pressed material to 1.3 Mg/m³, which may be connected with increase in the elastic unloading of material compressed to a greater degree. On these specimens, separation and traces of degradation in the surface layers of the polymer appeared. To avoid breakdown of the pressed material, in subsequent investigations, pressing was conducted at a pressure of 0.5–0.7 GPa, which ensures the production of a material with the highest density.

In spite of the high density, after explosive treatment, the pressed polymer does not possess a dense structure and requires sintering. The temperature range of subsequent sintering of the pressed material after explosive pressing was chosen on the basis of the heat resistance of Phenylone and amounted to 260–380°C.

The sintering of pressed Phenylone S2 and S3 of 3–4 mm thickness was conducted in the free state. As a result of analysing the results of the investigations carried out, a sharp reduction was found in the density of the material in the region of the glass transition temperature (260°C), from 1.32 to 1.15 Mg/m³ for S2 (Figure 2, curve 2) and to 1.17 Mg/m³ for S3 (Figure 3, curve 2). Further increase in temperature promotes softening of the polymers and an increase in density to maximum values of 1.29 Mg/m³ for S3 at sintering temperatures of 340–360°C, with a simultaneous increase in the microhardness of Phenylone to 320 MPa. The maximum density (1.30 Mg/m³) and microhardness (300 MPa) for Phenylone S2 are achieved at a temperature of 380°C. With increase in temperature above 380°C, the density of Phenylone S3 decreases sharply to values of less than 1 Mg/m³, and at the same time the hardness of the material falls to values of less than 180 MPa, which is caused by the start of degradation of the polymer.

In order to clarify the influence of the thickness of the pressed material on the process of making the polymer more dense, specimens of Phenylone S3 of 8–10 mm thickness with a density of 1.32–1.33 Mg/m³ were subjected to sintering. Sintering of pressed material was conducted at temperatures of 320–360°C in the free state and under pressure. The results of measuring density as a function of the heating temperature in the free state are presented in Figure 3 (curves 2 and 3), from which it follows that increase in the thickness of the pressed material leads to a sharper reduction in the density of

![Figure 1](image1.png)

**Figure 1.** Dependence of the density of Phenylone S3 on the shock-wave pressure

![Figure 2](image2.png)

**Figure 2.** Dependence of the density (1) and microhardness (2) of Phenylone S2 on the sintering temperature in the free state

![Figure 3](image3.png)

**Figure 3.** Dependence of the density (1, 2) and microhardness (3) of Phenylone S3 on the sintering temperature in the free state. 1 – specimen thickness 3–4 mm; 2 – specimen thickness 8–10 mm
the material in the glass transition region. Increase in temperature to 340–360°C promotes an increase in density to values that are slightly lower than the maximum values achieved for specimens of 3–4 mm thickness. Subsequent sintering of specimens at a temperature of 340–360°C under a pressure of 0.1–0.2 MPa, irrespective of their thicknesses, led to an increase in density to a maximum value of 1.3–1.32 Mg/m³.

It was of interest to examine the influence of the sintering temperatures on the mechanical properties of Phenylone with different treatments. In the work, the influence of the sintering temperatures of Phenylone S3 produced by static and explosive pressing was compared. From Figure 4 it can be seen that both materials acquire maximum hardness after heat treatment at a temperature of 360°C, the microhardness being higher for Phenylone produced by explosive treatment than for statically pressed Phenylone. Furthermore, in spite of the identical hardness of specimens in the region of the glass transition temperature, for material after explosive treatment the hardness increases more intensively, which confirms the better intermolecular interaction in the polymer, with possible crosslink formation, as in polymers previously investigated (UHMWPE, F-2M) [4]. As a result of this, the strength and deformation characteristics of the polymer change.

The results obtained were confirmed by microstructural and thermomechanical investigations. Figure 5 shows consolidation of particles occurs, with practically equal density, for a specimen produced by explosive pressing. After sintering, density increase occurs in specimens produced by static and explosive pressing, the structure of specimens after static pressing (SP) being characterised by greater porosity.

The results of TMA of Phenylone S3 (Figure 6) showed that explosive treatment leads to a 25–30°C shift in the softening points towards higher temperatures (curves 1 and 2). The thermomechanical curve of Phenylone S3 subjected to explosive pressing with subsequent sintering at 360°C is characterised by lower strains by comparison with a specimen produced by static pressing. The results of thermomechanical analysis of Phenylone S3 with different types of treatment are given in Table 1.

The changes in structure of Phenylone as a result of explosive loading, manifested by considerable shifts in the characteristic temperatures of physical processes, were confirmed by DTA and TGA. According to the results of analyses, the softening point and heat resistance of Phenylone are higher after shock-wave treatment. Explosive treatment led to a considerable shift in endo peaks towards higher temperatures. The structural changes in Phenylone during explosive treatment also affected the strength properties of the material.

Table 2 gives the physicomechanical properties of Phenylone S3 produced by explosive pressing in
comparison with the properties of Phenylone S2 produced by hot pressing [1]. From an analysis of the results of testing their properties it can be seen that Phenylone S3 produced by high-speed pressing hardly differs from Phenylone S2 produced by the more labour-intensive and time-consuming method, but in strength and hardness it is superior.

Thus, as a result of the conducted investigations it has been shown that the use of explosive pressing with subsequent sintering under pressure makes it possible to produce dense specimens of Phylenon of up to 10 mm thickness. High mechanical properties of pressed Phenylone is achieved by explosive pressing with a pressure of 0.5–0.7 GPa and subsequent sintering by the optimum regime: heating temperature 340–360°C for S3, heating temperature 380°C for S2, pressure 0.1–0.2 MPa.

REFERENCES


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<th>240°C</th>
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<th>280°C</th>
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<th>320°C</th>
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<tr>
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<td>1.0</td>
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Table 2. Physicomechanical properties of Phenylones obtained by explosive and static pressing

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<tr>
<td>Hardness, MPa</td>
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