

Shock-wave loading of W–PTFE–Al reaction materials based on nanoscale tungsten in cylindrical recovery ampoules

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Abstract. The effect of nanosized tungsten on the ignition of W–PTFE–Al compositions under shock-wave or thermal impact has been investigated. It has been found that ignition in the studied compositions occurs at 1000–1060 °C with rapid temperature increase to 1700 °C. The mechanism and localization of synthesis in the studied compositions under shock-wave loading in recovery ampoules depends on aluminum content. Compositions with low aluminum content (5–10 wt. %) ignite in low-pressure regions (upper part of the ampoule), whereas those with high Al content (20–30 wt. %) ignite in high-pressure regions (lower part of the ampoule). The 72W–18PTFE–10Al composition demonstrated the highest energy release, causing nearly complete ampoule destruction (70 % of length). The use of nanosized tungsten enabled shock-wave synthesis initiation in the 56W–14PTFE–30Al composition and revealed structural changes along the sample length. Following shock-wave loading of 64W–16PTFE–20Al and 56W–14PTFE–30Al compositions, the synthesized products underwent dispersion under atmospheric moisture exposure without phase composition changes.

Keywords: shock-wave loading; W–PTFE–Al; nanoparticles; reactive materials; combustion explosion.

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Ударно-волновое нагружение реакционных материалов системы W–ПТФЭ–Al на основе наноразмерного вольфрама в цилиндрических ампулах сохранения

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Аннотация. Исследовано влияние нанодисперсного вольфрама на инициирование горения составов системы W–ПТФЭ–Al при ударно-волновом или тепловом воздействии. Установлено, что воспламенение в исследуемых составах инициируется при 1000...1060 °C с интенсивным ростом температуры до 1700 °C. Механизм и локализация синтеза при ударно-волновом нагружении исследуемых составов в ампулах сохранения зависит от содержания алюминия. Составы с низким содержанием алюминия (5–10 мас. %) инициируются в области с низким давлением (в верхней части ампулы), тогда как составы с высоким содержанием Al (20–30 мас. %) в области с высоким давлением (в нижней части ампулы). Состав 72W–18ПТФЭ–10Al продемонстрировал

наибольшее энерговыделение, так как привел к почти полному разрушению ампулы (70 % длины). Использование нанодисперсного вольфрама позволило инициировать синтез ударно-волновым нагружением в составе 56W–14ПТФЭ–30Al и установить изменение структуры по длине образца. В результате ударно-волнового нагружения составов 64W–16ПТФЭ–20Al и 56W–14ПТФЭ–30Al полученные продукты синтеза подвержены диспергированию под действием атмосферной влаги без изменения фазового состава.

Ключевые слова: ударно-волновое нагружение; W–ПТФЭ–Al; наночастицы; реакционные материалы; тепловой взрыв.

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1. Introduction

The synthesis of functional materials using shock-wave loading (SWL), particularly with preservation ampoules, is a promising research direction [1–4]. A preservation ampoule is a container for holding the test powdered material, intended for subsequent processing with intense shock waves and preservation of the resulting sample for further analysis. This method provides short-term exposure (on the order of microseconds, 10^{-6} s) to the material, creating extreme pressures and temperatures within it [5–11]. During this time interval, under plastic deformation and interparticle friction, the surface of the powder particles becomes activated, accompanied by the destruction of oxide films. As a result, a strong metallic framework forms, and adhesion at interphase boundaries is enhanced, ultimately enabling the production of composites with improved physico-mechanical properties (strength, density, corrosion resistance, and friction coefficient) [5–8]. One of the key features of SWL is its ability to initiate exothermic chemical reactions in materials [12, 13]. Thus, in reactive systems, such as aluminum with polytetrafluoroethylene (Al–PTFE), shock-wave impact can trigger intense heat release, potentially leading to the initiation of a self-propagating high-temperature synthesis mode and subsequent destruction of the preservation ampoule [11, 14].

Reactive materials (RM) include powder compositions that are inert under normal conditions but capable of intense exothermic reactions when initiated by thermal or shock-wave impulses [1, 4]. This category encompasses compositions for self-propagating high-temperature synthesis (SHS) [13], thermite [4], metallic [15], and metal-polymer systems [4]. Among them, metal-polymer compositions, particularly aluminum with polytetrafluoroethylene (Al–PTFE), attract special interest. This composition exhibits a unique set of properties, such as high sensitivity to shock-wave impact, high gas release, and high specific mass

energy (up to $8.5 \text{ MJ} \cdot \text{kg}^{-1}$), which is more than twice the energy of TNT [15]. PTFE in this system serves as a source of oxidizers (fluorine, carbon), plasticizer, and protective barrier against atmospheric influences. The combination of metallic and polymeric phases in the composite allows merging the advantages of both components: the strength, electrical and thermal conductivity of aluminum with the low friction, chemical resistance, and processability of the polymer [5]. This makes the Al–PTFE system promising for creating RM based on it.

Despite the advantages of the Al–PTFE system, its practical application is limited by low density and insufficient strength [4]. To address these issues, the composition is modified with high-density metallic additives such as tungsten, nickel, etc. [14, 17]. The study [17] demonstrates that the introduction of these additives significantly enhances the composite's strength characteristics. However, a negative trend is observed: increasing the concentration of strengthening additives leads to a decrease in the overall heat release of the reaction and sensitivity to shock-wave loading, due to the reduced proportion of energetically active components (Al and PTFE) in the mixture. One approach to enhancing the reactivity of high-density additives is increasing the particle dispersion [18].

Special preservation ampoules with cylindrical or flat shapes are used for experimental studies of reactive materials by shock-wave loading [8–10]. The choice of ampoule configuration is determined by the required loading mode, as different pressure and deformation gradients form in the center and periphery of the sample, leading to heterogeneity in the properties of the synthesized material [5]. Thus, the geometry of the preservation ampoule is a key parameter determining the spatial distribution of the composite properties after SWL.

The aim of this paper is to study the dependence of the W–PTFE–Al system sensitivity to shock-wave loading in cylindrical preservation ampoules on the dispersion of the tungsten used.

2. Materials and Methods

2.1. Preparation of raw materials and thermal explosion techniques

For the preparation of reactive materials of the W–PTFE–Al system, powders of nanoscale tungsten (≤ 500 nm), polytetrafluoroethylene (PTFE) of the fluralit grade (≤ 5 μm), and aluminum of the ASD-1 grade (≤ 50 μm) were used. Prior to use, all powders were subjected to moisture removal in a drying oven at a temperature of 150°C for 12 hours. Powder mixing was carried out in a gravitational mixer for 3 hours. The drum rotation speed was 30 rpm, and the ratio of the mass of grinding media to the mass of the powder mixture was 5:1.

The ignition temperature of the compositions under study was determined by the thermal explosion method [19]. For this purpose, cylindrical samples with a diameter of 3 mm and height of 1.4–1.7 mm were used. The sample was placed in a boron nitride crucible, which was installed directly on the junction of a WR5/20 type thermocouple with a thickness of 50 μm . The crucible with the sample and thermocouple was placed on a graphite heater. The heating rate of the samples was $60\text{--}80^\circ\text{C}\cdot\text{s}^{-1}$. Experiments were conducted in a reactor with an inert argon atmosphere (purity $\geq 99.998\%$).

Shock-wave impact on the compositions under study was carried out using cylindrical preservation ampoules. Structurally, the ampoule was a steel cylinder 100 mm high with an outer diameter of 16 mm. Both ends of the cylinder had threads for fixing the conical upper and flat lower plugs. Eight cylindrical samples with a diameter and height of 10 mm, pre-pressed to a relative density of 0.97–0.99, were placed inside the ampoule.

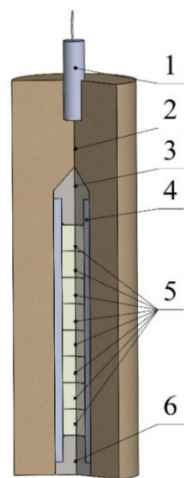


Fig. 1. The scheme of shock-wave loading of recovery ampoules with the studied compositions:

1 – detonator; 2 – explosive;
3, 6 – upper and lower plugs; 4 – pipe; 5 – samples

The use of eight samples ensures dense filling of the internal volume of the ampoule, allowing for uniform material density throughout the height. The gap between individual samples was practically absent, as the upper conical cap, when tightened, pressed against the last (upper) sample, ensuring their tight fit to each other. The ampoule with samples was centered inside a cylindrical assembly with a diameter of 50 mm and height of 150 mm. Ammonite grade 6ZhV was used as the explosive (EX). All assembly components (detonator, EX charge, and ampoule) were installed coaxially with each other (Fig. 1).

2.2. Analytic methods

The phase composition of the products obtained after shock-wave synthesis was studied by X-ray phase analysis on a DRON-3M diffractometer. Diffraction patterns were recorded with an angular step of 0.02° and an exposure time of 1 s. Quantitative analysis of the synthesis products was determined by the corundum number method. This method involves quantitative determination of the phase composition of a multicomponent powder mixture by comparing the intensities of diffraction peaks of the studied phases with a standard – corundum ($\alpha\text{-Al}_2\text{O}_3$), without the need to construct calibration curves using reference mixtures.

3. Results and Discussion

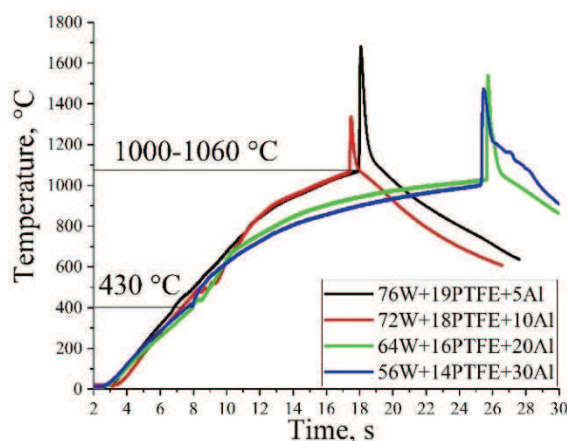
For thermodynamic calculations in the THERMO program, data for tetrafluoroethylene (C_2F_4), were used, since polytetrafluoroethylene (PTFE) is a polymer based on this monomer. The standard enthalpy of formation of C_2F_4 $\Delta H = -658.5 \text{ kJ}\cdot\text{mol}^{-1}$ was taken from the reference [20]. In studies [19, 21], optimal compositions for the W–PTFE–Al system from microdispersed powders were established based on calculations (Table 1). Table 1 presents the results of thermodynamic calculations performed using the THERMO program [22], including values of adiabatic combustion temperature (T_{ad}), as well as the proportion and phase composition of the resulting products. Previously, in studies [19, 20], it was established that in composition No. 1 with low aluminum content, self-sustaining combustion does not initiate upon local heating, but synthesis initiates under shock-wave loading, whereas in composition No. 4 with high aluminum content, self-sustaining combustion initiates upon local heating, but synthesis does not initiate under shock-wave loading.

Table 1. Characteristics of the compositions

No	Mass content, wt. %			Theoretical density, g·cm ⁻³	T_{ad} , °C	Condensed product, wt. %	Gas product, wt. %
	W	PTFE	Al				
1	76	19	5	6.93	2777	75 (W ₂ C)	16 (AlF ₃); 3 (WF ₃)
2	72	18	10	6.4	2866	74 (W ₂ C)	11 (AlF ₃); 7 (AlF); 5 (AlF ₂)
3	64	16	20	5.55	1710	66 (W ₂ C)	29 (AlF)
4	56	14	30	4.91	1382	57 (W ₂ C)	24 (AlF)

Measurements of the ignition temperature of the W–PTFE–Al system based on nanodispersed tungsten in the thermal explosion mode showed that the reaction proceeds with high intensity in all compositions (Fig. 2). The ignition intensity is characterized by the time between the onset of ignition and reaching the maximum temperature and is about 0.1 s. Initiation of reactive mixtures was observed upon reaching the temperature range of 1000–1060 °C with subsequent increase to 1400–1700 °C. The noted ignition temperature threshold significantly exceeds the phase transition points of individual components – melting of aluminum and decomposition of PTFE, confirming the heterogeneous interaction mechanism involving solid, liquid, and gaseous reagents. Experimentally recorded peak temperature values were significantly lower than the calculated adiabatic parameters. This discrepancy is explained by the combined effect of the low sample mass, contributing to heat losses, and the significant volume of gaseous products released, absorbing part of the reaction energy.

Figure 3 shows preservation ampoules after shock-wave loading. Deformation and integrity violation of all ampoules confirm that synthesis was initiated in all compositions under shock-wave loading (Fig. 3).

**Fig. 2.** Thermograms of the heating process of the studied compositions

Upon initiation of the 76W–19PTFE–5Al composition, the upper plug was ejected with partial destruction of the ampoule wall (approximately 30 % of its length), as shown in Fig. 3a. The 72W–18PTFE–10Al composition exhibited a more intense reaction with ejection of the upper plug and partial destruction of the ampoule wall (approximately 70 % of its length) (Fig. 3b). This composition demonstrated the maximum energy release among the studied systems. Upon initiation of the 64W–16PTFE–20Al composition, ampoule depressurization occurred with characteristic expansion of its upper part: the diameter increased by 12 % (Fig. 3c). The reaction of the 56W–14PTFE–30Al composition had the least impact on the ampoule: its hermeticity was preserved, but there was an increase in diameter in the lower part by approximately 6 % (Fig. 3d).

The distribution of damage regions in the ampoules allows assessing the relative sensitivity of the studied compositions to shock-wave impact. For the 76W–19PTFE–5Al and 72W–18PTFE–10Al compositions, destruction was localized exclusively in the upper part of the ampoule, while the lower section retained the original geometry without visible deformations (Fig. 3a, b). As is known [8], under cylindrical loading, maximum pressure is realized in the lower part. Initiation of compositions in the upper part of the ampoule indicates their high sensitivity to shock-wave loading, which is consistent with the results described in [19].

Deformation and destruction of ampoules with 64W–16PTFE–20Al and 56W–14PTFE–30Al compositions occurred predominantly in their lower part (Fig. 3c, d). Localization of the reaction near the ampoule bottom indicates reduced sensitivity of these compositions to shock wave impact. The use of nanodispersed tungsten powder proved to be a key factor that enabled initiation of the synthesis reaction in the 56W–14PTFE–30Al composition, whereas in [19] this composition based on microdispersed tungsten did not initiate.

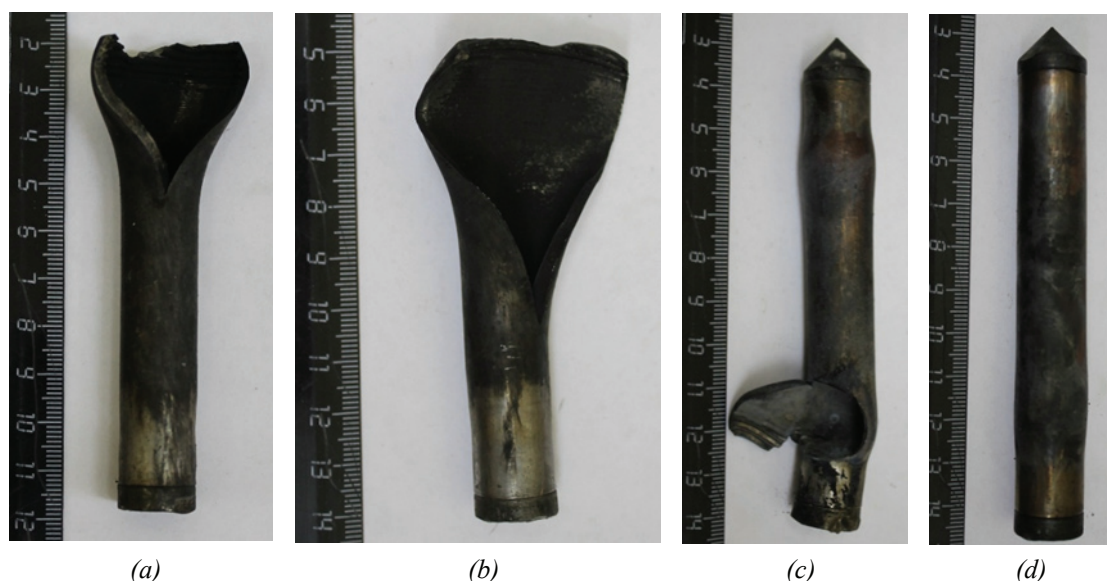


Fig. 3. Ampoules after shock-wave loading:

a – 76W–19PTFE–5Al; *b* – 72W–18PTFE–10Al; *c* – 64W–16PTFE–20Al; *d* – 56W–14PTFE–30Al

Ampoules with 64W–16PTFE–20Al and 56W–14PTFE–30Al compositions were cut along the detonation propagation direction of the explosive. In the ampoule with the 64W–16PTFE–20Al composition, the product was preserved in the upper part, which is associated with ampoule destruction and ejection of most of the sample (Fig. 4*a*).

Reaction products were collected from four sections: beginning (I), middle (II), end (III), and directly from the ampoule walls in the middle part (IV). Their X-ray phase analysis was conducted, with results shown in Fig. 5. Products from sections I, II, and III had identical composition consisting of tungsten carbides (hexagonal WC – 35–38 wt. %, W_2C 22–26 wt. %) and tungsten aluminide (monoclinic Al_4W 32–36 wt. %). The product collected from the ampoule walls (IV) mainly consisted of aluminum fluoride (rhombohedral AlF_3 52–54 wt. %), while WC (12–15 wt. %), W_2C (12–15 wt. %), and Al_4W (13–16 wt. %) were present in approximately half the amount. Thus, gaseous aluminum fluoride (AlF_3) is released from the sample and condenses on the ampoule walls. The synthesis product formed large solid pieces, but upon exposure to air, it disintegrated into powder state (Fig. 4*b*). This effect is associated with moisture absorption from air by aluminum fluoride, forming $AlF_3 \cdot 3H_2O$ in the synthesized product, which leads to its volume expansion [23, 24]. The resulting powder was pressed into tablets and dried until the tablet weight stopped decreasing, with a weight loss of 1–2 %. Dried

tablets, when exposed to air for several hours, absorbed up to 1 % moisture and disintegrated similarly to the products from the preservation ampoule (Fig. 4*b*). Thus, disintegration is caused by moisture sorption from air.

During shock-wave loading of the ampoule with the 56W–14PTFE–30Al composition, synthesis occurred, which did not happen in the composition based on microdispersed tungsten [20]. The internal part of the synthesized sample had several large pores in sections I and II, as well as fine pores along the entire sample length (Fig. 6*a*).

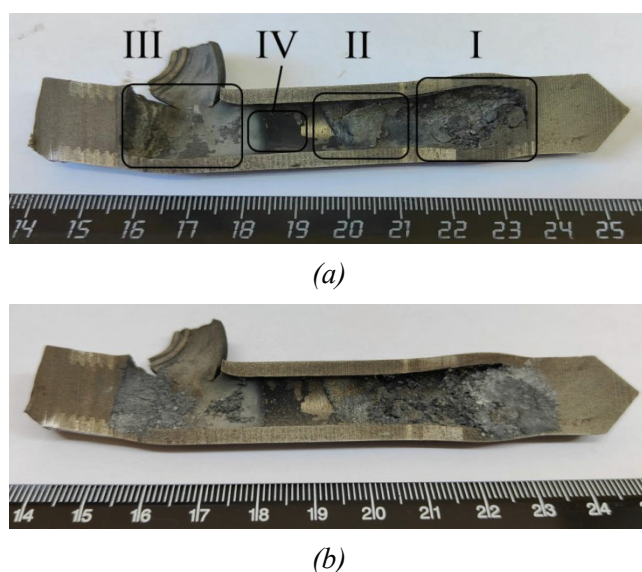


Fig. 4. The longitudinally-sectioned ampoule with composition 64W–16PTFE–20Al after shock-wave loading: *a* – after the experiment; *b* – 170 hours later

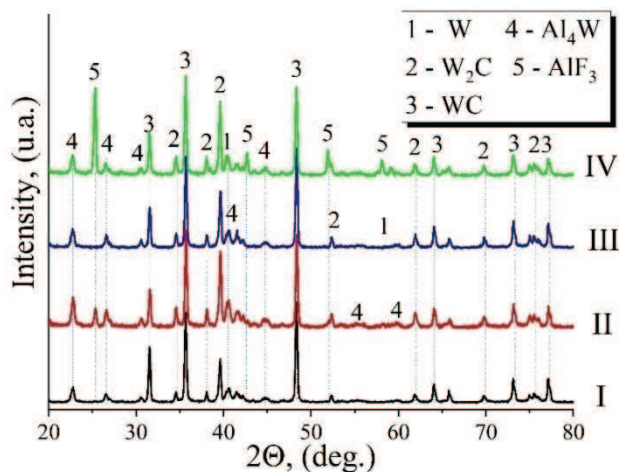
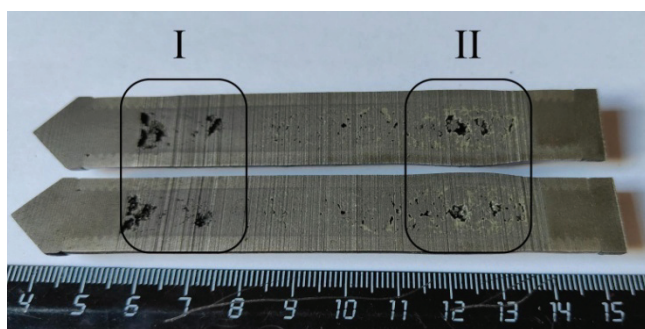
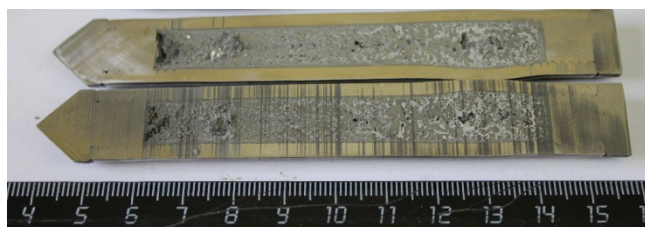


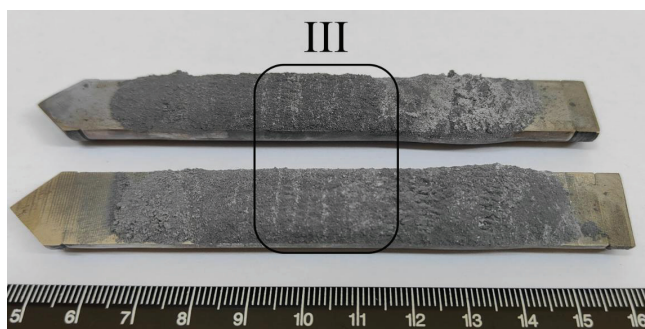
Fig. 5. XRD patterns of the synthesis products from the 64W-16PTFE-20Al ampoule after shock-wave loading



(a)



(b)



(c)

Fig. 6. The longitudinally-sectioned ampoule with composition 56W-14PTFE-30Al after shock-wave loading:
a – after the experiment; b – 24 hours later;
c – 720 hours later

The sample can be divided into three regions. The first region – 25 mm long – consists of a light-gray fine-grained phase distributed in a dark-gray phase. The second region – 20 mm long – features a characteristic 45° inclination angle of the light-gray and dark-gray phases relative to the sample axis. The third section, approximately 35 mm long, consists of a coarse-grained light-gray phase distributed in a dark-gray phase. The observed significant gradient of synthesis conditions along the sample length is due to the convergence of compression and reflection shock waves in the lower part of the preservation ampoule [19]. The shock waves also converge toward the sample center, creating uneven pressure from periphery to center, which leads to the formation of regions with different reaction intensity [1].

The resulting synthesized product represented a solid sample along the entire ampoule length (Fig. 6a), but after 24 hours, the beginning and end of the sample began to disintegrate (Fig. 6b). After more than 700 hours, the solid synthesized product disintegrated into powder state (Fig. 6c). To determine air moisture sorption by the synthesis products of the 56W-14PTFE-30Al composition, the powder was dried. The mass loss after drying was 2.0–2.5 %. The weight of tablets after exposure to air for 12 hours increased by 1 %, and edge crumbling of the tablet and disintegration occurred similarly to the products from the preservation ampoule.

Figure 7 shows the results of X-ray phase analysis of synthesis products from the ampoule with the 56W-14PTFE-30Al composition, taken from sections I and II, as well as after moisture absorption from section III (Fig. 6c). The higher intensity of

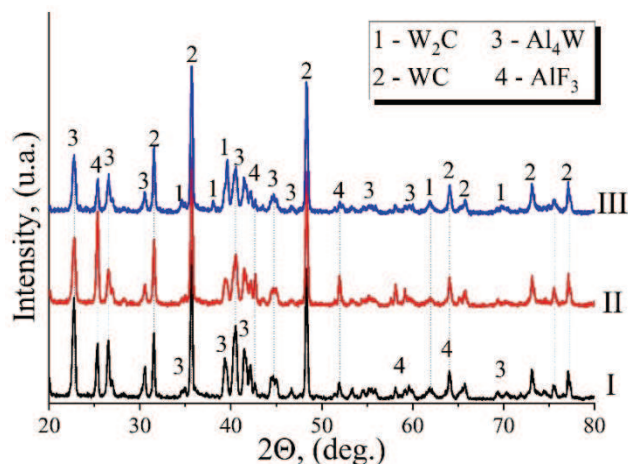


Fig. 7. XRD patterns of the synthesis products from the 56W-14PTFE-30Al ampoule after shock-wave loading

AlF₃ peaks in section 2 correlates with the observed significant deformation of the ampoule in this area, indicating more intense reactions with formation of volatile products specifically in the lower part of the sample. The intensity of AlF₃ peaks in the synthesis products after moisture absorption (section III) decreases, but the phase composition is identical to the synthesis products from sections II and III of the sample before moisture absorption.

4. Conclusion

As a result of the conducted studies, the parameters of thermal explosion in the W–PTFE–Al system using nanodispersed tungsten were determined. For all studied compositions, the reaction is characterized by high rate of occurrence. The time interval between ignition at 1000–1060 °C and reaching peak temperatures in the range of 1400–1700 °C is about 0.1 s. The use of nanodispersed tungsten enabled initiation of synthesis under shock-wave loading in all compositions. The 76W–19PTFE–5Al and 72W–18PTFE–10Al compositions are highly sensitive to shock-wave loading, as they initiate in the region with relatively low pressure. The 64W–16PTFE–20Al and 56W–14PTFE–30Al compositions demonstrate low sensitivity. The use of nanodispersed tungsten made the synthesis products hygroscopic. The main phases in all synthesis products are tungsten carbides (WC, W₂C) and tungsten aluminide (Al₄W). The hygroscopicity of the synthesis products led to their disintegration into powder state. Disintegration is associated not with phase composition change, but with the physical process of water sorption.

As a result of shock-wave loading of the 56W–14PTFE–30Al composition in a cylindrical preservation ampoule, formation of three morphologically distinct regions was revealed. The observed heterogeneity indicates the existence of a significant gradient of synthesis conditions (temperature, pressure, and reaction duration) along the sample length. The 72W–18PTFE–10Al composition demonstrated the most intense energy release under shock-wave loading, as evidenced by the greatest ampoule destruction (70 % of length).

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6. Conflict of interests

The authors declare no conflict of interest.

Конфликт интересов

Авторы заявляют об отсутствии конфликта интересов.

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