

# Phase and Structural Transformation of Polyacrylonitrile Fiber during Two-Stage Thermal Stabilization

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**How to cite this paper:** Fazlitdinova, A.G. and Tyumentsev, V.A. (2020) Phase and Structural Transformation of Polyacrylonitrile Fiber during Two-Stage Thermal Stabilization. *Journal of Materials Science and Chemical Engineering*, 8, 54-63.  
<https://doi.org/10.4236/msce.2020.811005>

**Received:** October 27, 2020

**Accepted:** November 24, 2020

**Published:** November 27, 2020

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## Abstract

The influence of two-stage isothermal treatment on the change in the linear dimensions of the fiber, the average sizes of the coherent scattering regions, the texture and phase composition of the polyacrylonitrile fiber in the process of isothermal thermal stabilization is considered by the methods of dilatometry and X-ray diffraction analysis. It is shown that preliminary short-term heat treatment at a lower temperature affects the process of structural transformations of the polyacrylonitrile fiber material and the formation of a new highly dispersed phase of the thermally stabilized fiber.

## Keywords

Polyacrylonitrile Fiber, Thermal Stabilization, X-Ray Diffraction Analysis, Coherent Scattering Regions

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## 1. Introduction

Carbon fibers (CF), made on the basis of polyacrylonitrile (PAN) fibers, in comparison with viscose and pitch, occupy a dominant position in the market [1] [2]. High strength, thermal and chemical resistance, high modulus of elasticity in combination with low density determine their widespread use as a reinforcing material in high-performance carbon-polymer and carbon-carbon composites [3] [4] [5]. The elastic-strength properties of carbon fibers are determined by the structure, which begins to form already at the stage of manufacture of the precursor PAN fibers [6]. In industry, CF are obtained by preliminary oxidative thermal stabilization at temperatures below 300°C in an air atmosphere and subsequent high-temperature (up to ~3000°C) carbonization of PAN fibers in an inert atmosphere [7] [8].

It is believed that the stage of oxidative thermal stabilization of PAN fiber is one of the most important, significantly affecting the final structure and properties of carbon fibers [4] [9] [10] [11]. Thermomechanical treatment of PAN fibers initiates chemical reactions of dehydrogenation, cyclization, and oxidation, resulting in the formation of polymer regions with a ladder structure [2] [4] [10] [11]. In this case, a thermally stabilized fiber nanostructure is formed in the PAN fibers (in the X-ray diffraction patterns, as the intensity of the (010) diffraction peak decreases, the intensity of the new maximum increases at  $2\theta \sim 25.5$  degrees, due to the formation of coherent scattering regions (CSR) of a new phase [8] [9]). It is believed that this phase spatially inherits the structure of the original fiber [12] [13]. The process is accompanied by the removal of gaseous products, such as  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{HCN}$ ,  $\text{NH}_3$ , etc. [12] and significant shrinkage of the material [12] [14].

The conditions of thermal stabilization affect the regularities of the transition of the polyacrylonitrile fiber into a thermally stabilized state. It was shown that the formation of a polymer with a ladder structure develops more actively in the presence of oxygen. The increased oxygen content in the surface layer of the fiber determines its structural inhomogeneity over the cross section [13] [15] [16] [17]. Preliminary  $\gamma$ -Ray irradiation of PAN fiber stimulates a more active diffusion of oxygen into the volume of fiber, as a result of which, in the course of thermal stabilization, fibers homogeneous over the cross section are formed [18]. Irradiation of the PAN precursor with an electron beam also promotes more active formation of the structure of the thermally stabilized fiber [19]. It is also noted that a certain dose of  $\gamma$ -Ray irradiation stabilizes the texture of PAN in the process of thermal stabilization, as a result of which the elastic-strength properties of the resulting CF increase noticeably [20].

Optimal drawing of PAN fiber during thermal stabilization stimulates dehydrogenation and oxidation reactions [12]. The pulling stress promotes the orientation of the molecular and ladder structure, which is inherited by the CF structure during the subsequent high-temperature treatment [12] [13]. In the process of drawing, at the initial stage of thermal stabilization, the degree of crystallinity increases, an increase in the CSR's size and texturing of PAN is observed [14] [21] [22]. The effect of the temperature of isothermal stabilization and pulling load on the kinetics of the phase transition, as well as the process of changing the texture and CSR's average size of the PAN fiber, was investigated in [14] [22] [23] by X-ray diffraction analysis.

The aim of this work was to systematically study the structure of samples differing in the duration of preliminary heat treatment at a lower temperature, obtained successively at different stages of the formation of a thermally stabilized fiber under conditions of thermal stabilization. X-ray diffraction analysis was chosen as the research method, which allows obtaining data on changes in average sizes and interplanar distances of CSRs of the initial and newly formed phases, as well as changes in the texture of PAN fiber during thermal stabilization.

## 2. Experimental

The studies were performed on PAN fibers made with dimethyl sulfoxide (methyl acrylate content 5 wt%). Metal rings are attached to the ends of the PAN fiber harness for fixation in the installation for thermal stabilization. The initial length  $\ell_0$  of each fiber harness was recorded, which corresponded to the distance between the rings. Subsequently, the length of the PAN fiber was measured  $\ell = \ell_0 + \Delta\ell$  as a function of the time of isothermal treatment. Preliminary isothermal treatment of fiber harness containing 500 filaments was carried out at 200°C for 5, 15, and 30 minutes. Subsequent isothermal thermal stabilization was carried out at a temperature of 265°C. Heat treatment was carried out in an air atmosphere at a constant pulling load of 0.6 g/tex, the time of moving the fiber into the furnace was 1 - 2 seconds, and the accuracy of maintaining the temperature was  $\pm 1^\circ\text{C}$ .

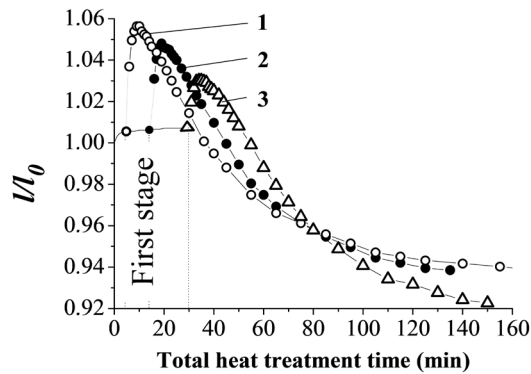
Structural studies of the fibers were performed using a D8 ADVANCE X-ray diffractometer (filtered  $\text{CuK}\alpha$  radiation). To provide the Bragg-Brentano self-focusing condition for the diffracted beam, the entire analyzed volume of the fiber was placed in the form of a thin (0.1 - 0.2 mm) layer of filaments. The profiles of the (010) PAN diffraction peak, formed by the CSR, oriented relative to the fiber axis at an angle  $\varphi$  from 0 to  $\pm 5$  degree (scan step one degree), were analyzed using the Origin software. The interplanar distance and CSR's average sizes were calculated from the center of gravity and the integral width of the peak. To characterize the texture of the PAN fiber, we used the Z value, the width (in degrees) at half height of the dependence of the maximum intensity of the (010) diffraction peak on the angle  $\varphi$  of the CSR's orientation relative to the fiber axis, in the interval of which  $\sim 75\%$  of the material under study is oriented [22].

## 3. Results and Discussion

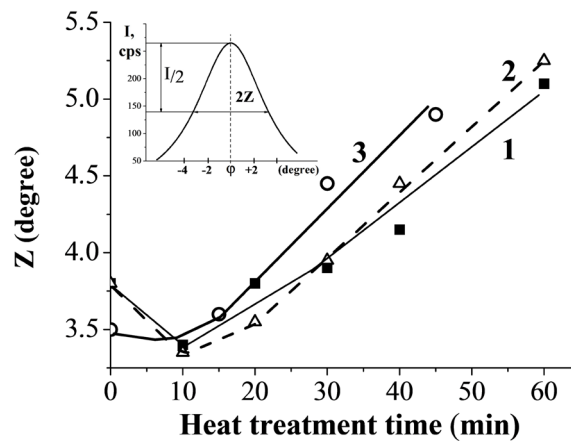
At the initial stage of preliminary thermomechanical treatment of PAN fibers at 200°C (in the first 3 min), plastic flow of the material is observed, the relative elongation  $\ell/\ell_0$  of the fiber is  $\sim 1\%$ , **Figure 1**. Continuation of isothermal treatment at this temperature for up to 30 min has practically no effect on the increase in the linear dimensions of the fiber.

The texture parameter Z of the PAN fiber material is 3.95 degree. During the preliminary treatment, the value of Z decreases to 3.8 degree (samples No. 1, 2) and 3.5 degree (sample No. 3), **Figure 2, Table 1**. The average sizes of the CSR, oriented at an angle  $\varphi = 0$  degrees, after heat treatment for 5, 15 and 30 minutes. Increased by  $\sim 13$ ,  $\sim 12$  and  $\sim 12\%$ , respectively, compared to the original PAN fiber (see **Table 1**).

At the initial stage of the subsequent thermal stabilization of the obtained samples No. 1, 2, and 3 of the PAN fiber at a higher temperature of 265°C, an active increase in the length of the fiber is also observed. The  $\ell/\ell_0$  value of a fiber that has undergone preliminary treatment for 5 min increases by  $\sim 5\%$ , **Figure 1**.



**Figure 1.** Relative change in the length of the PAN fiber during thermomechanical treatment at 200°C for 5, 15, 30 minutes (First stage), and subsequent thermal stabilization at 265°C (preliminary isothermal treatment at 200°C for: 1 - 5 min, 2 - 15 min, 3 - 30 min).



**Figure 2.** The relationship between the duration of thermal stabilization at a temperature of 265°C and the parameter Z, characterizing the change in the texture of the polyacrylonitrile fiber (preliminary isothermal treatment at 200°C for: 1 - 5 min, 2 - 15 min, 3 - 30 min). The inset shows the curve of the change in the intensity of the (010) diffraction peak with an increase in the orientation angle of the CSR relative to the fiber axis at an angle  $\varphi$  from 0 to  $\pm 5$  degree for the precursor PAN fiber.

**Table 1.** Changes in the interplanar distance and sizes of CSR oriented parallel to the fiber axis, as well as the texture parameter of the PAN fiber during thermal stabilization at 265°C.

No. Sample	Thermomechanical treatment modes		Structure of PAN			Structure of thermally stabilized fiber	
	Temperature, °C	Duration of heat treatment	$d_{010}$ , Å	$L_{010}$ , nm	Z, degrees	d, Å	L, nm
		Precursor PAN fiber	5.288	13.3	3.95	-	-
1	200	5 minutes	5.220	15.0	3.8	-	-
		10 minutes	5.261	16.7	3.4	-	-
	265	20 minutes	5.236	16.2	3.8	3.38	1.7
		30 minutes	5.249	14.0	3.9	3.39	2.3

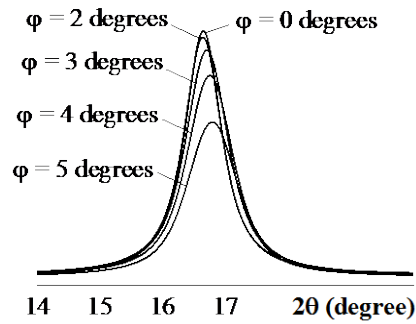
## Continued

		40 minutes	5.281	10.4	4.15	3.46	2.5
		60 minutes	5.258	6.2	5.0	3.46	1.8
		120 minutes	5.283	5.2	5.1	3.47	1.6
		180 minutes	-	-	-	3.48	1.5
2	200	15 minutes	5.270	14.9	3.8	-	-
		10 minutes	5.233	17.1	3.35	-	-
		20 minutes	5.226	15.5	3.55	3.33	1.8
	265	30 minutes	5.270	11.3	3.95	3.44	2.2
		40 minutes	5.267	10.5	4.45	3.46	2.1
		60 minutes	5.288	7.5	5.25	3.43	2.0
		120 minutes	5.331	4.1	-	3.48	1.8
3	200	30 minutes	5.307	14.9	3.5	-	-
		15 minutes	5.285	15.4	3.6	-	-
		30 minutes	5.316	12.3	4.45	3.38	3.2
	265	45 minutes	5.331	8.9	5.0	3.43	2.7
		60 minutes	5.321	5.6	-	3.41	1.8
		120 minutes	-	-	-	3.43	1.5

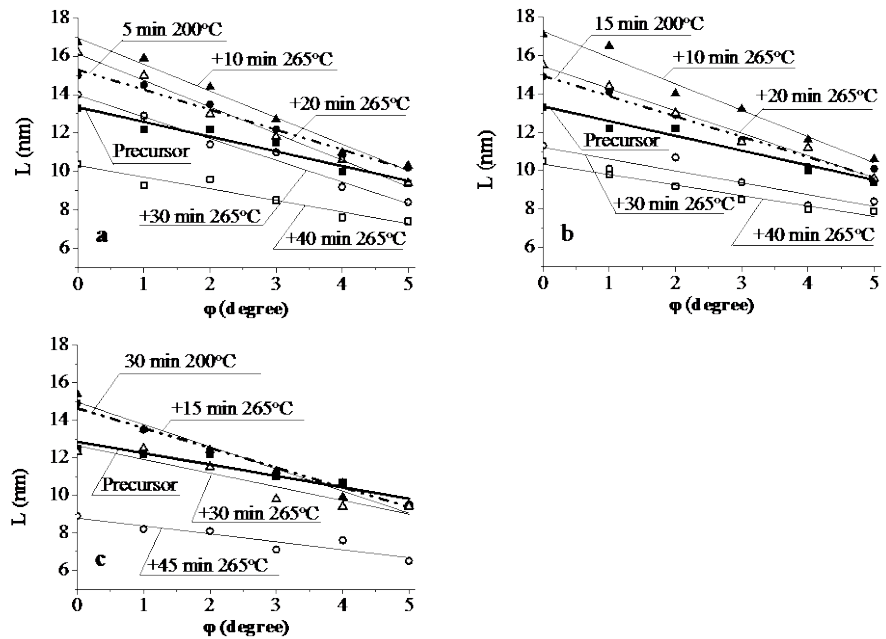
The relative elongation of the fiber that underwent preliminary heat treatment at 200 °C for 15 and 30 min is ~4% and ~2.5%, respectively. Polyacrylonitrile fiber material for samples No. 1 and 2 becomes more textured, the Z parameter decreases to 3.4 and 3.35 degrees, see **Table 1**. At the same time, after heat treatment of sample No. 3 for 15 min, the parameter Z increased from 3.5 to 3.6 degrees.

Continuation of thermal stabilization is accompanied by shrinkage of fiber. The  $\ell/\ell_0$  value at the end of thermal stabilization is ~0.95 and ~0.92 for fibers that have undergone preliminary heat treatment for 5, 15 minutes and 30 minutes, respectively. The texture of the polyacrylonitrile fiber is destroyed. This process develops somewhat more actively in a fiber that has undergone preliminary heat treatment for 30 minutes; after thermal stabilization for 45 minutes, the value of the parameter Z reaches to 5 degrees, **Figure 2**.

Let us consider the effect of preliminary thermomechanical treatment on the change in the CSR's average size  $L_{010}$  of a polyacrylonitrile fiber in the process of thermal stabilization. In contrast to the structure of the PAN fiber described in [14], the crystalline component of the initial PAN fiber material is represented only by CSR, the average size of which is ~13 nm (there is no highly dispersed component, the CSR's size of which is ~1.5 nm), see **Table 1**. As the angle  $\varphi$  of the CSR orientation relative to the fiber axis increases from 0 to 5 degrees an increase in the integral width of the (010) peak is observed, **Figure 3**. The calculated dimensions of  $L_{010}$  decrease by ~30%, **Figure 4**.



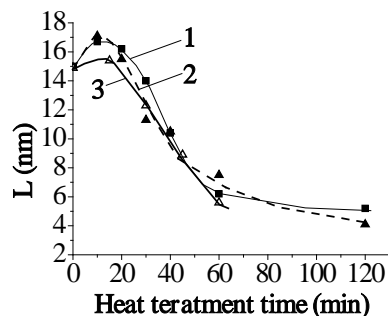
**Figure 3.** The profiles of (010) diffraction peak CSR of the precursor PAN fiber oriented at an angle  $\varphi$  of 0 to 5 degrees.



**Figure 4.** Dependences of the CSR's average sizes  $L_{010}$  of polyacrylonitrile fibers on the orientation angle relative to the fiber axis  $\varphi$  at different stages of thermal stabilization. Preliminary thermomechanical treatment of PAN fibers at 200°C for (a) 5, (b) 15, and (c) 30 minutes.

Preliminary heat treatment at 200°C for 5, 15 and 30 minutes (Samples No. 1, 2, and 3, respectively) resulted in almost the same (by ~13% at  $\varphi = 0$  degree) increase to ~15 nm of average sizes  $L_{010}$ , see **Table 1**, **Figure 4**. In this case, as can be seen in the presented dependences, the  $L_{010}$  sizes of the CSR oriented at an angle  $\varphi = 5$  degree to the fiber axis did not practically increase.

After thermal stabilization of samples No. 1 and 2 prepared in this way at 265°C for 10 minutes, the CSR's average sizes  $L_{010}$  of the PAN fiber increased to 16.7 and ~17.1 nm. At the same time, the  $L_{010}$  sizes of sample No. 3, which underwent thermal stabilization for 15 minutes, increased by only ~3%, to ~15.4 nm (**Figure 5**). A further continuation of thermal stabilization causes a gradual decrease in the average  $L_{010}$  values of all the samples studied. After thermal stabilization of samples No. 1 and 2 for 40 minutes, and sample No. 3 for 45 minutes,



**Figure 5.** Change in the  $L_{010}$  CSR's sizes of samples No. 1, 2 and 3 of PAN fibers during thermal stabilization at a temperature of 265°C (preliminary isothermal treatment at 200°C for: 1 - 5 min, 2 - 15 min, 3 - 30 min).

the average size  $L_{010}$  of the CSR oriented parallel to the thread axis ( $\varphi = 0^\circ$ ) decrease to  $\sim 10.5$  and  $\sim 9.5$  nm, respectively.

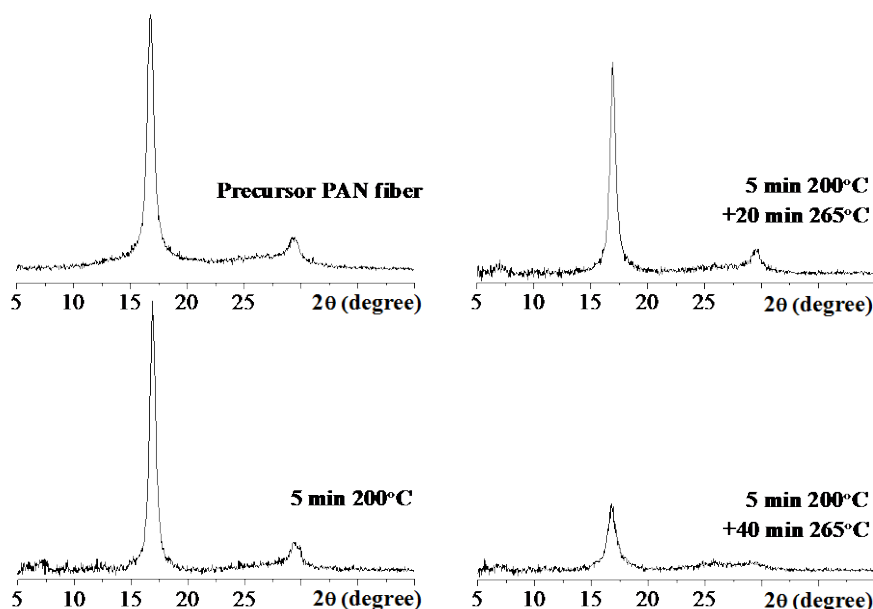
The observed changes in the texture and dispersion of the CSR of polyacrylonitrile fiber are due to the appearance of a new phase (on the X-ray diffraction patterns of the samples, a broad diffraction peak is formed at  $2\theta \sim 26$  degree, the intensity of which increases with an increase in the duration of thermal stabilization), **Figure 6**. It can be assumed that chemical and structural changes in polyacrylonitrile develop in local microvolumes and are stimulated by the thermal effects of cyclization and oxidation.

As a result, a new phase is formed in the local microvolumes of the CSR of polyacrylonitrile fiber, the average sizes of the coherent scattering regions of which are of the order of 2 nm and practically do not change with the duration of thermal stabilization (an increase in the amount of phase is observed).

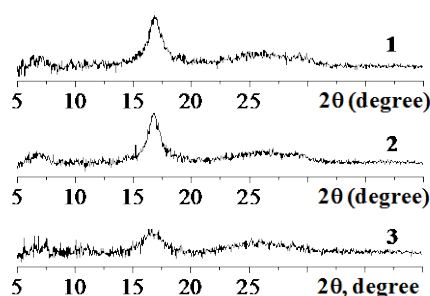
The interplanar distance of the new phase is comparable to that of  $d_{002}$  of turbostratic carbon. After thermal stabilization of samples No. 1, 2 and 3 of PAN fibers at 265°C for 60 minutes, a weak peak of 010 PAN and a rather intense wide maximum at  $2\theta \sim 26$  degrees phase of the thermally stabilized fiber are present in the X-ray diffraction patterns, **Figure 7**. The ratio of the number of phases "PAN/thermally stabilized fiber", estimated from the ratio of the peak areas at  $2\theta \sim 17$  degree and  $\sim 26$  degree, is 54/46,  $\sim 54/46$  and  $\sim 40/60\%$ , respectively.

Thus, preliminary thermomechanical treatment at 200°C stimulates the improvement of the crystal structure and texture of PAN. With the duration of such a treatment  $\leq 15$  minutes, at the initial stage of the subsequent thermal stabilization at a higher temperature, a significant stretching of the fiber is observed under the action of a pulling load. There is also an increase in the CSR's average size and an increase in the texture of PAN. In the process of further thermal stabilization, a new structure of the thermally stabilized fiber is formed, the CSR orientation of which should inherit that of the PAN CSR.

An increase in the duration of preliminary heat treatment at 200°C to 30 minutes (sample No. 3) led to the fact that at the initial stage of stabilization at a higher temperature (in the first  $\sim 5$  minutes) the relative elongation  $\ell/\ell_0$  of the



**Figure 6.** Fragments of X-ray diffraction patterns of precursor PAN fibers, fiber that has undergone preliminary heat treatment at 200 °C for 5 minutes and subsequent stabilization at 265 °C for 20 and 40 minutes.



**Figure 7.** Fragments of X-ray diffraction patterns of samples No. 1, 2, and 3 of PAN fibers that have undergone thermal stabilization at 265 °C for 60 minutes (preliminary isothermal treatment at 200 °C for: 1 - 5 min, 2 - 15 min, 3 - 30 min).

fiber turned out to be two times less compared with sample No. 1. In the process of further thermal stabilization of the PAN fiber that has undergone prolonged preliminary heat treatment, a more active dispersion and destruction of the texture of the polymer material is observed.

#### 4. Conclusions

Preliminary short-term thermomechanical treatment at 200 °C is accompanied by lengthening PAN fibers, an increase in the CSR's size, and an increase in the texture of the material.

At the initial stage of the subsequent isothermal stabilization at a higher temperature, the drawing of the PAN fiber, the growth of the CSR's size, and the texturing of the material are resumed. The most active increase is the size of the PAN CSR oriented parallel to the fiber axis.



With an increase in the duration of the preliminary thermomechanical treatment of PAN fibers at 200 °C from 5 to 30 minutes, the fiber drawing at the initial stage of thermal stabilization at 265 °C is reduced and the transformation of the material into the structure of the thermally stabilized fiber is activated.

### Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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