

Effect of cryogenic treatment on the fracture toughness of aircraft aluminum alloy 7075

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Abstract. Influence of three types of the treatment on fracture toughness of the Al-7075 alloy was investigated in this study. Commercial Al-7075 alloy in the solid solution heat-treated condition was processed by hardening with post-cryogenic deformation treatment and PVD deposition titanium and copper coatings. The fracture toughness was estimated with using macroscopic and microscopic approaches. The conditions for the coincidence of the fracture toughness estimates between brittle fracture mechanics and the photometric analysis of structural images (PHASI) methods were achieved. The highest fracture toughness was obtained by applying hardening, cryogenic compression, ageing and deposition of the Ti-coating, leading to dispersion particles precipitation.

1. Introduction

The development of strengthening treatments for high-strength aluminum alloys is a big challenge of the Aircraft Industry. Modern strengthening treatments of the high-strength alloys include combination of the several strengthening mechanisms at the same time: a) alloying by chemical elements which are able to provide solid-solution and dispersion hardening effects; b) heat treatment and deformation in a wide range of temperatures [1-8].

There is limitation for the high-strength materials associated with increasing possibility of the brittle fracture in the result of complex multi-stage strengthening treatments. Among many mechanical properties of the high-strength aluminum alloys the fracture toughness is one of the most important due to exceptional structurally sensitiveness. The fracture toughness is able to be a criterion limiting high-strength materials application [9].

The fracture toughness is determined by the methods of fracture mechanics as special fracture criteria for three modes of destruction: the opening mode (Mode I), the in plane shear mode (Mode II) and out-of-plane shear mode (Mode III) [10,11]. These criteria are called the stress intensity factors (SIF) and are denoted as K_{Ic} , K_{IIc} and K_{IIIc} . (The SIF depends significantly on the sample thickness). In case of plane strain minimum SIF is typical for large sample thicknesses. For plane stress minimum SIF is typical for small sample thicknesses. In real constructions the high-strength materials are used to produce thin-walled shells, therefore, the K_{Ic} is of more interest for the evaluation of fracture toughness.

It should be noted that fracture mechanics determine the limiting conditions for the equilibrium of bodies with macroscopic cracks. The conditions of microcracks nucleation and mechanisms of their growth up to macroscopic dimensions are not possible for fracture mechanics methods. However, understanding the structural mechanisms of cracks nucleation and growing is a necessary requirement



to solve the problems of materials strengthening. Thus, the study of elementary mechanisms of destruction at the micro-level opens new possibilities for correct estimation of SIF.

2. Experimental procedure

Experiments were performed on plates (6-mm thick, 20 × 20 mm) of cold rolled 7075 aluminum alloy, which was produced using standard industrial practices. The chemical composition of this alloy is shown in Table 1.

Table 1 Chemical composition of 7075 aluminum alloy (wt. %)

Alloy	Al	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Other Each	Others Total
7075	Rem	0.40	0.50	2.0	0.30	2.9	0.28	6.1	0.20	0.05	0.15

Every sample had electric spark notch with 4.0 mm length and 0.4 mm width cut along the vertical axis of sample symmetry.

Specimens were treated by using three types of technological operations, which are shown in Table 2.

Table 2 Technological operations of the Al-7075 samples

№	Treatment	Applied operations	Treatment parameters			
			D (mm)	Δh (μm)	T _h / t, (°C/min)	T _a / t, (°C/min)
1	Original state	Cold rolled condition	-	-	-	-
2	Cryogenic strengthening + ageing	Hardening + cryogenic compression + ageing	-	-	-	45/30
3	Cryogenic strengthening + Cu-coating	Hardening + cryogenic compression + ageing+ Cu-coating	11	0,18	450/30	45/30
4	Cryogenic strengthening + Ti-coating	Hardening + cryogenic compression + ageing+ Ti-coating	11	0,18	450/30	45/30

D – strengthening zone diameter; Δh – depth of the pressure in strengthening zone; T_h- hardening temperature; T_a - ageing temperature; t- operation duration.

Hardening and ageing processes were performed in vacuum atmosphere with using quartz ampoules. Cryogenic compression was conducted in liquid nitrogen fluid in T= - 196 °C. Titanium and copper coatings were deposited on the one surface of the samples in the vacuum universal system VUP-5 (USSR). Electric spark notches were cut after completion of treatment operations for each sample.

Specimens were loaded by inserting a wedge into the notch according to Hillman technique [12] with 1 mm/ min rate on an Instron-3382 (USA) test machine. The loading scheme of the samples is shown in Figure 1

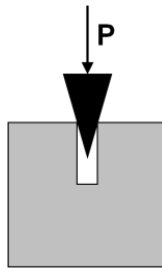


Figure 1. Hillman loading technique

Figure 2 shows the loading curve of the ordinary state sample in the coordinates load (P) – time (t).

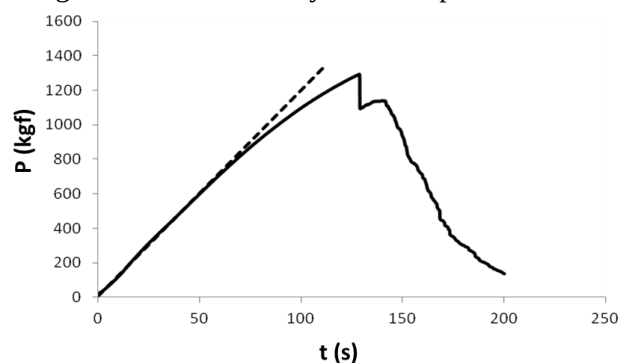


Figure 2. Load – time curve of ordinary state sample Al-7075

During loading samples surfaces images were recorded using video camera Panasonic HDC-HS60. The images were selected with 10s, 5s and 2s time steps and then analyzed using photometric analysis of structural images (PHASI) [13], which had been developed by high-voltage microscopy laboratory team at Institute of Metallurgy and Material Science.

PHASI is a method based on the comparative analysis of the digital images using spectrums brightness of visible light reflection from the surface before loading and in the process of loading. PHASI is a program-analytical platform that allows to dye the chosen parts of the spectrum in different colors, to move these colors to the digital image and determine spectrum density of any part of the image. Each spectrum parameter of images matches with physical parameter of changing, which were observed during experiment. On the basis of this data, calibration graph was constructed, where spectrum parameters transform to 1. local physical characteristics of materials, 2. level of degradation material as result of physical influence, including kinetics of this process.

Coordinates of PHASI comparative analysis: $p(I)$ - spectrums brightness density of the reflection - intensity of reflection (I). Spectrums brightness density of the reflection $p(I)$ was calculated using the formula:

$$p(I) = \frac{n(I_i)}{N} \quad (1)$$

$n(I_i)$ – images pixels with intensity I_i ; N - overall amount of the pixels.

Intensity of reflection (I) was measured in the conventional units of the linear scale, where 0 – surface condition of the total visible light absorption; 1 – surface condition of the total visible light reflection.

Figure 3 shows comparison of the sample surface structures and spectra of the visible light reflection before start loading (a) and in 10s loading (b).

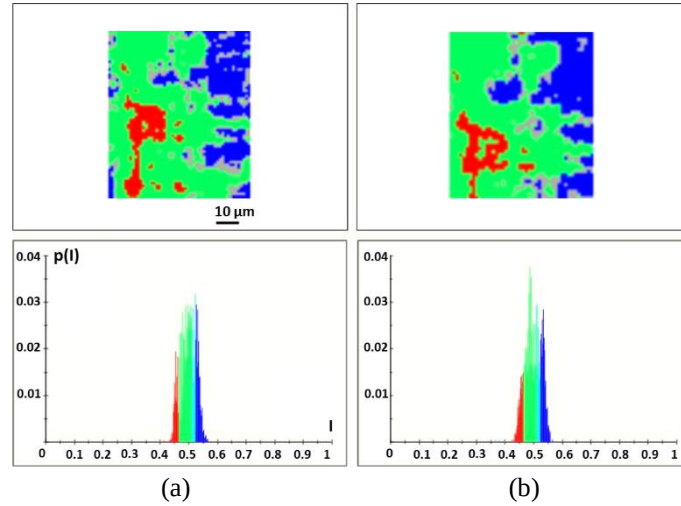


Figure 3. Sample surface structures and spectra of the visible light reflection before start loading (a) and in 10s loading (b), obtained by PHASI method

The PHASI allows to estimate the degree of the material structural modification (D_s) and describe it as:

$$D_s = \frac{S_i(t) - S_i(0)}{S_{max} - S_{min}} \quad (1')$$

$S_i(t)$ - spectral curve area of the i fragment captured at moment t ; $S_i(0)$ - spectral curve area of the i fragment captured at start of loading; S_{max} and S_{min} - maximum and minimum spectral curve areas at the time interval $[0, t]$.

Using this criterion, the value of the load corresponding to the beginning of the crack propagation was determined. After that the stress intensity factor (SIF) was determined. For the SIF definition, the standard methods of the fracture mechanic and PHASI method were applied. According to fracture mechanic method SIF was determined using finite-element method [14] as:

$$K_{Ic} = \frac{P}{tW^{1/2}} \times f(a) \quad (2)$$

P – load of the crack initiation moment; t – sample thickness; W – geometry parameter; $f(a)$ – coefficient, defined as:

$$f(a) = (2+\alpha) \cdot (0,8072 + 8,858\alpha - 30,23\alpha^2 + 41,088\alpha^3 - 24,15\alpha^4 + 4,951\alpha^5) \cdot (1-\alpha)^{-3/2} \quad (2')$$

$\alpha = a/W$, a - The length of a crack from tip of a crack to the loading axis.

PHASI allows to estimate energy of the light reflection from the sample surface (E_s) in conventional units. This energy is numerically equal to the spectral curve area. According to [15], the body emission energy is related to its internal energy (U) as:

$$E_s = AU \quad (3)$$

A – coefficient describing the possibility of the spontaneous body light emission;

Coefficient A was determined from the equation (3) by plugging into it elastic energy value for the set moment of time and the elastic energy value in the conventional units defined by PHASI method.

This energy balance equation can be described as:

$$\Delta U = U_{\sigma} - U_0 = \frac{\sigma^2}{2E} \quad (4)$$

Where U_{σ} - internal energy for the sample under the load; U_0 - internal energy for the sample before the load; E - Young's modulus (modulus of elasticity). σ – stress

Thus, strain can be calculated as:

$$\sigma = \sqrt{2AE\Delta U} \quad (5)$$

To estimate strain in the tip of the crack the notch area of the sample was divided into three fragments bordered to each other (figure 4)

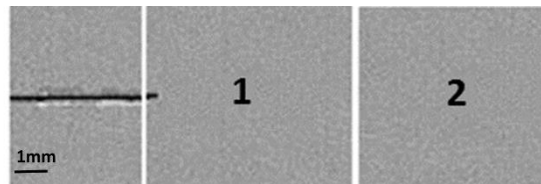


Figure 4. Sample fragments with notch for PHASI analysis: 1 - the microfragment closest to the notch, 2 - the fragment at the edge of the sample, without a notch.

The stress in the center of the fragments was determined from the equation (5). Fracture toughness values (K_{Ic}) were calculated from [11]:

$$K_I = \sigma_y \sqrt{2\pi r} \quad (6)$$

Where r - distance between tip of the crack and center of the fragment

3. Results and discussion

Figure 5 shows Internal energy – time (t) curve of the 7075 alloy after technological operation №2 (hardening + cryogenic compression + ageing).

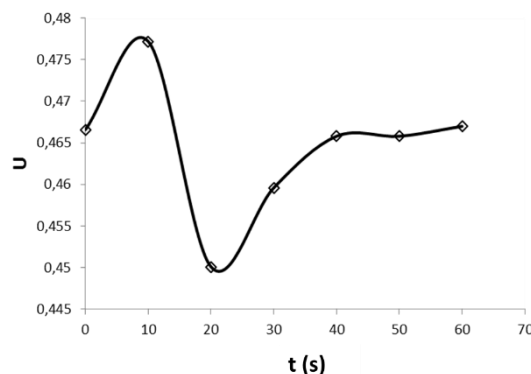


Figure 5. Internal energy – time curve of the 7075 alloy after technological operation №2

Figure 6 shows material structural modification (D_s) – time (t) curve of the 7075 alloy after technological operation №2 (hardening + cryogenic compression + ageing).

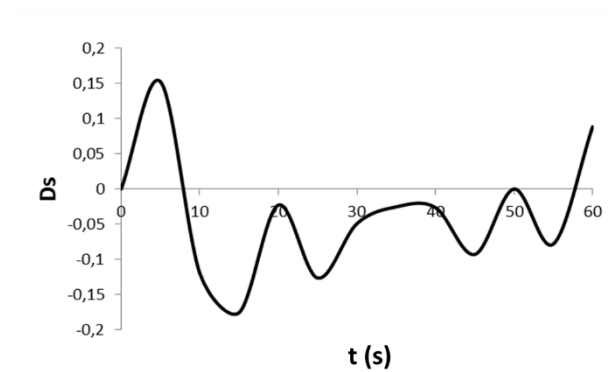


Figure 6 Material structural modifying– time curve of the 7075 alloy after technological operation №2

PHASI of the sample surface showed that crack grows by consistent change of the three main stages: the first - stage of the plastic zones forming in the fronts, the second - the stage of the notch opening, the third – the stage of the crack propagation. Figure 7 shows material structural modifying (D_s) – distance from crack tip (r) curve of the 7075 alloy after technological operation 2. The maximum D_s value is fixed at some distance from the crack notch $r = 1.65$ mm. It means that microstructure damage happens when three modes of destruction are in action (Mode I+ Mode II + Mode III) more precisely, under plane strain conditions [12, 16].

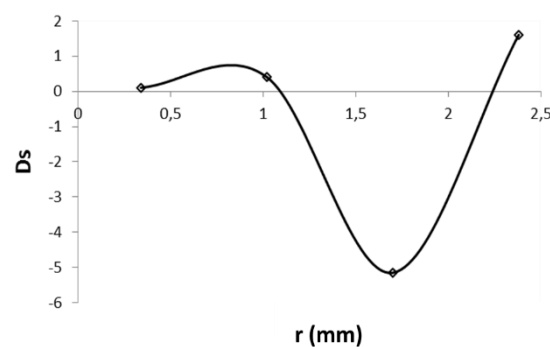


Figure 7 Material structural modifying (D_s) – distance from crack tip (r) curve of the 7075 alloy after technological operation 2

Figure 8 shows the stress intensity factor (K_{Ic}) – time (t) curves. The K_{Ic} criteria are calculated by using fracture mechanic method (equation 2) and PHASI method (equation 6). The calculated dependencies shown in Figure 8 are approximated by analytical formulas, which are given in Table 3.

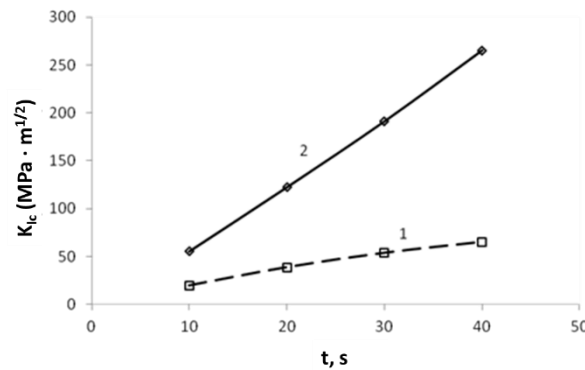


Figure 8. Stress intensity factor (K_{Ic}) – time (t) curves of the 7075 alloy after technological operation 2, $r=1,165$ mm. 1 – K_{Ic} were calculated using fracture mechanic method; 2 – K_{Ic} were calculated using PHASI method.

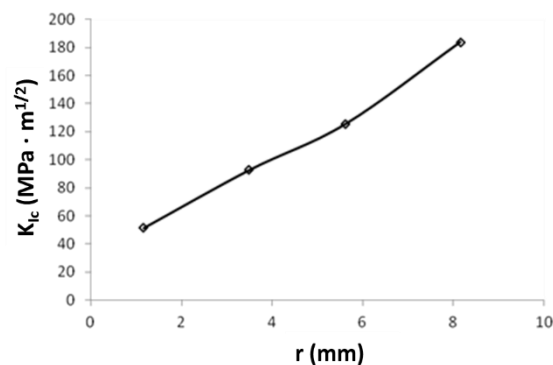


Figure 9. Stress intensity factor (K_{Ic}) – distance from crack tip (r) curves of the 7075 alloy after technological operation 2, $t=40$ s.

Table 3 Analytical formulas approximating calculated dependencies $K_{Ic} = f(t)$

Equation for K_{Ic}	Approximating equation	Significance test
(2)	$K_{Ic} = 2,7277 \cdot t^{0,8702}$	0,9943
(6)	$K_{Ic} = 6,9875 \cdot t^{-16,063}$	0,9995

In this research [17] the condition of equality of the K_{Ic} obtained by using fracture mechanic and PHASI methods was determined. These values for alloy 7075 are the following: $K_{Ic} = 49.1$ MPa for fracture mechanic method and $K_{Ic} = 51.3$ MPa for PHASI method. The experiment showed that $K_{Ic} = 49.1$ MPa is reached within 20 s of the load. the same value of the fracture toughness determined using PHASI method was reached within 9s of the load. Both dependences $K_{Ic} - t$ are practically linear dependences on time. The results of experiments and K_{Ic} calculating for all technological operations are presented in table 4.

According to the data in Table 4 the highest fracture toughness was obtained by applying technological operation №4, which includes hardening, cryogenic compression further ageing and deposition of the Ti-coating, which lead to dispersion particles precipitation.

Table 4 Results of the calculating K_{Ic} for all technological operations

№	Treatment	t (s)	P (kg)	Δl (mm)	K_{Ic} (MPa · m ^{1/2})	
					fracture mechanic	PHASI
1	Original state	13,3	165,6	0,67	14,7	40,4
2	Cryogenic strengthening + ageing	29,2	207,84	1,77	18,5	115,8
3	Cryogenic strengthening + Cu-coating	27,2	208,32	1,22	18,5	108,3
4	Cryogenic strengthening + Ti-coating	50	357,22	0,23	32,8	143,1

4. Conclusions

Investigation of the influence of the three types of treatment on the fracture toughness of the aluminum alloy 7075 process has been performed in this study. The results show that most perspective treatment for fracture toughness increasing includes hardening, cryogenic compression further ageing and deposition of the Ti-coating. This technology allows to increase fracture toughness by 2-4 fold (depending on the evaluation method) compared to the original state of 7075 alloy. The developed microscopic PHASI method is able to estimate fracture toughness using compact samples and to reach appropriate correlation with macroscopic approach of the fracture mechanic method. PHASI is a promising technique with potential for industrialization and therefore needs to be further investigated in any aspect.

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