



## Spherical particles formation under biaxial cyclic loading due to mesotunneling effect

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**ABSTRACT.** Fatigue fracture surfaces of Al-based alloys with fatigue striations pattern and such wear debris pattern as spherical particles were investigated fractographically, on the bases of the OG'e spectroscopic analysis. The sequence of events during fatigue crack edges opening was discovered when the elliptical or spherical shapes of wear debris build up on the fracture surface in crosspieces between mesotunnels under mode III of mode I fatigue crack opening because of volume rotation. The cause of black colour of places with fretting patterns on the fracture surfaces of Al-based alloys is discussed.

**KEYWORDS.** Mesoscopic fatigue fracture; Fractography; Spherical particles; OG'e spectroscopy; Rotation plastic deformation.

### INTRODUCTION

A metal with a growing crack represents an open dynamic system, which is far from equilibrium [1]; the system is exercising a series of sequential transitions from one to another stability state and the continued energy exchange with the environment. An open system evolves by passing through the critical states, referred to as the bifurcation points, to, alternatively, a stability or instability condition [2]. As long as the system experiences fluctuations, it cannot avoid instability immediately before a bifurcation point. The newly activated processes of damage accumulation develop or, alternatively, die out, depending on whether the system is able to the self-organized absorption of energy in the ways that shift the construction element toward a greater stability, i.e. longer life-time.

The evolution of an open system is commonly discussed in the terms of microscopic, mesoscopic, or macroscopic scale levels [3]. The first is relevant to the effects on the atomic spacing; the second, to the behaviour of atomic ensembles, and the third, to the creation of bulky space structures. As cyclic loading of a construction element is continuing, mechanisms of damage accumulation replace one another sequentially for the three scale levels, each starting and keeping on for a certain time [4].

The microscopic (Stage I) and mesoscopic (Stage II) scale levels of crack growth are in common with one another as concerns the subjects of forming shear lips at the free surface of the work-piece [5], mesoscopic tunnelling (or holes) of crack, or combining shear and cleavage in the metal when subjected to uniaxial tension, Fig.1.

Isolated regions of the failed metal, formed all along the crack front, are stretched in the crack-growth direction and separated by unbroken crosspieces. Mesotunnels (or holes, see Fig.1) are formed by shear during Stage I, and the crosspieces can fail, during Stage I, by the type-III shear or by growing a crack from one to other tunnel just like growing the tunnels themselves. However, rotational instability of deformation and fracture of the inter tunnel crosspieces may



become the case at Stage II (Fig. 2, (b), (c), and (d)). Changing from the shear- to rotation-type instability in crosspiece is associated with further complication of the way in that energy is being absorbed in the material before fracture.

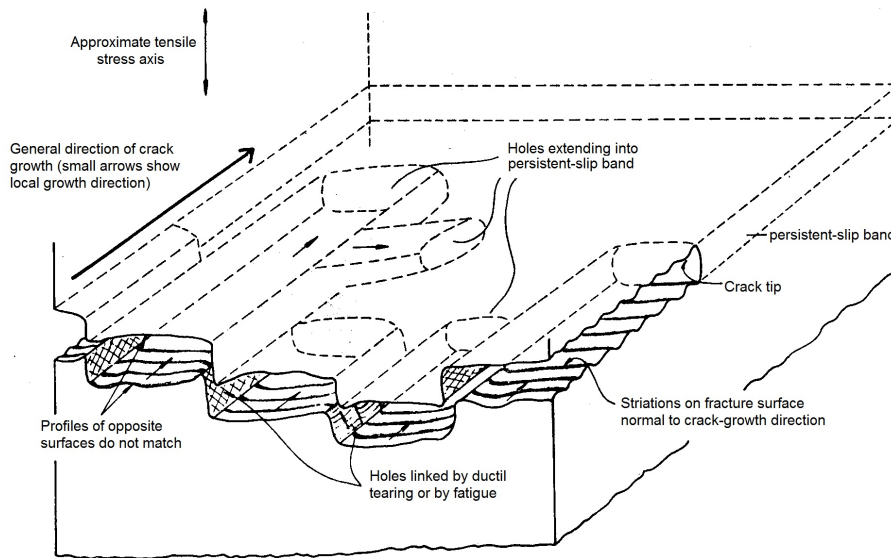


Figure 1: Schema of metals fatigue cracking with simultaneously holes (mesotunnels) formation and crosspieces between them failure because of shearing or fatigue striation creation [6].

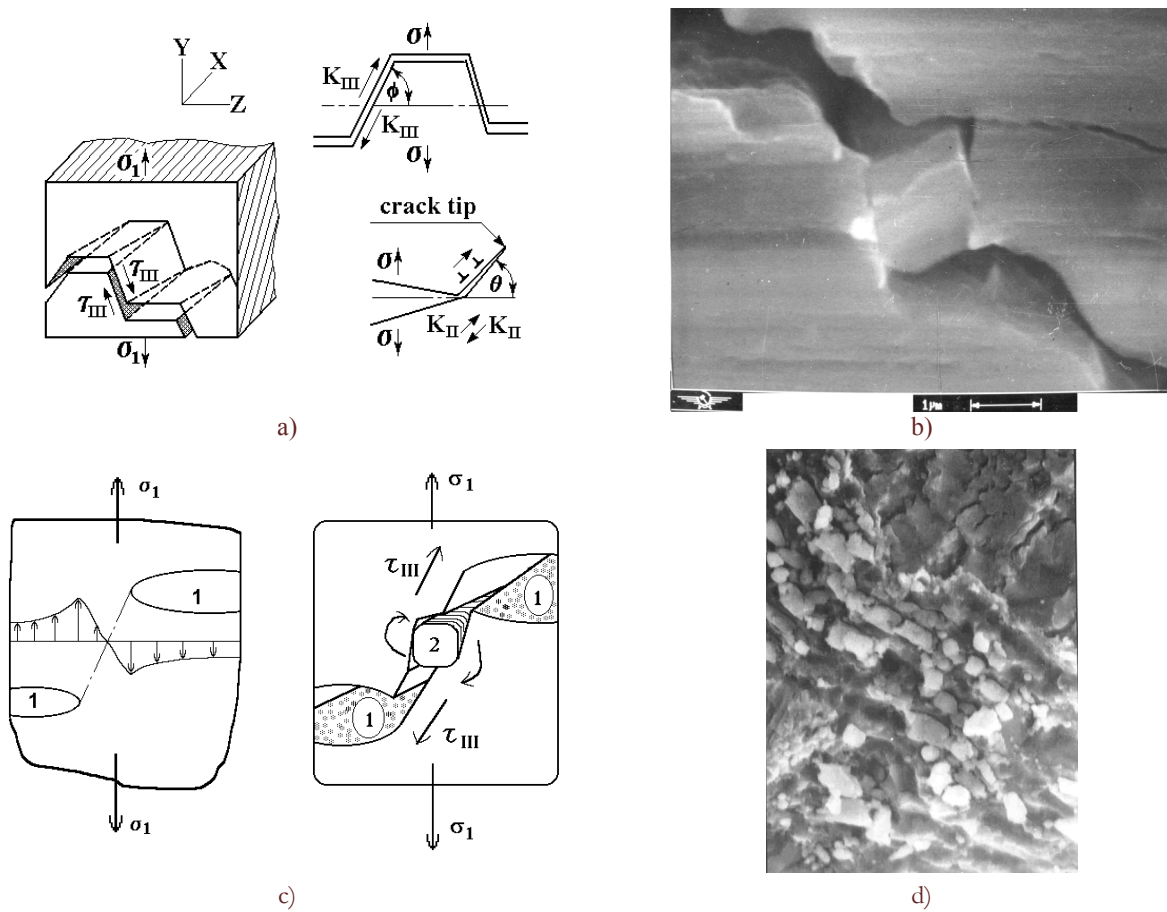


Figure 2: Combined mode-I, II and III opening of a fatigue crack under uniaxial stretch condition with (a) mesotunnels- 1, (b),(c) material rotations in crosspieces (2) between mesotunnels, and (d) cascade of oriented in chain cylindrical, ellipsoidal and spherical particles.



It was fractographically confirmed [7] that, at the microscopic scale level, the crack-growth behaviour is quite sensitive to the microstructure of the materials, and dislocation slip is dominated. Ivanova V.S. and Shanyavskiy A.A [4] have shown that the crack growth, at Stage I, in mesotunnels is associated with the development of slip: multiple-slip traces, slip steps or extrusion sites can be seen at the background of the pseudo-striations pattern. The fatigue crack propagation is quite fast in the mesotunnels at the Stage I. Consequently, the system experiences a self-organized transition to more complicated ways of energy absorption by the material, subjected to deformation, in which new free surface is being formed for meso-tunnels; this transition to the mesoscopic scale level (Stage II) occurs once the critical conditions at the crack tip were created [8]. The energy-absorption process becomes more complicated since the rotation effects are dominating in the deformation and fracture of the material at the meso-tunnel tip.

Fractographic analyses of fatigue surfaces attest to Stage II (tensile Mode I) striation formation [4, 7], following Stage I crack growth. The dramatic decrease in crack growth acceleration between the two Stages is strongly exhibited by the kinetic ( $da/dN \propto \Delta K$ ) diagram for long cracks at the point of change in slope (or deviation) is witnessed under a regular cyclic loading condition. Therefore, a self-organized transformation from one form of energy absorption to another is occurring near to the crack tip. The shear mode of material separation (the mode II process) is the dominant mechanism of metal fracture below this deviation point whereas the opening mode (mode I process) is dominant above this point.

This paper presents an analysis of the mechanisms involved in the formation of spherical particles at the mesoscopic scale level based on a rotation effect and the shear sliding process for aluminium based alloys. Both mechanisms were investigated fractographically, and, also, on the bases of the OG'e spectroscopy analyses.

Let be consider a process of spherical particles formation in crosspieces between meso-tunnels.

### SPHERICAL PARTICLES DUE TO MODE III OF MODE I FATIGUE CRACK GROWTH

Spherical particles wear formation under various cyclic loads conditions is well-known phenomenon [9-11]. They were discovered in compositions of wear debris are formed during rolling contact fatigue [9]. Further these particles were looking on the fretting surface [10, 11].

Fatigue cracks development in components or specimens is accompanied by processes of wear debris patterns formation on the fatigue surface because of crack edges interaction [4, 12-14]. The main idea for the interaction based on the process [13], which due to the mode II shearing of the mode I cracks growth under external tension loading for the near threshold fatigue cracks development. Rewelding occurs at the contact points across the crack as  $\Delta K_I$  falls [14]. Wear debris become detached from both fatigue surfaces during the rewelding and tearing processes. The leading role of the mode  $K_{II}$  in contact points across cracks front have to be attracted to discuss the first stage of the fatigue crack growth because of the shearing mechanism which directed to the roughed surface formation. Suresh S. and Ritchie R.O [13] performed the roughness-model to calculate effective stress intensity factor for fatigue cracks growth under Modes ( $K_I + K_{II}$ ).

The Mode II crack growth in specimens from Fe- and Al-based alloys was modelled under compressive cyclic loads, and spherical particles on the fatigue surface were shown [15]. There were places with wear debris of the black colour on the fatigue fracture surfaces for Al-based alloy. Two sizes for different particles shapes were discovered: (10...40)  $\mu m$ , and smallest than 10  $\mu m$ . The smallest particles were dominant. Small particles were often associated with small sockets and wear tracks, where the particles have been removed using replicating tape. The higher contrast suggested that they might contain a significant proportion of oxide. Their higher contrast in micrographs and their apparently greater hardness than the matrix tend to suggest that they contained a large amount of oxide. But it had not yet been possible to determine their exact composition.

Various models [9-11] were discussed in the paper [15] and it was shown that they cannot explain a mechanism of the small particles formation.

The well-known model [10] of wear particles formation by adhesive wear processes which trapped them in cavities in the sliding surface and became soothed by burnishing processes, can explain big particles formation only. According to the model, spherical particles to be anticipated in slow uniaxial sliding, in fretting and within cracks of a material being fatigued.

Below results of the spherical particles fractographic analysis for fatigued specimens from Al-based alloys is discussed, and OG'e analysis uses to explain a mechanism of their creation during fatigue cracks growth.



### Fractographic analysis

Spherical particles fractographic analysis was performed on the fatigue surface of specimens from Al-alloys tested earlier under various cyclic loads conditions [4]: bending of cylindrical bars with rotation; uniaxial tension of prismatic specimens with tension decreasing so that a crack retardation for the semi-elliptically shaped cracks took place, then spherical particles formation process is occurred after the retardation; biaxial tension-compressive of cruciform specimens with semi-elliptically shaped cracks; biaxial overloads of cruciform specimens for through-thickness cracks. Materials compositions used for the investigation have shown in Table 1.

	Mg	Si	Cu	Mn	Fe	Zn	Ti	Cr	Al
AVT	0.82	0.67	0.38	0.24	0.48	0.19	0.1	0.2	remained
D1T	0.73	0.62	0.46	0.77	0.36	0.21	0.06	-	remained
D16T	1.4	0.3	4.2	0.65	0.42	0.15	0.07	-	remained
AK6T	0.47	1.13	2.25	0.42	0.57	0.24	0.08	-	remained

Table 1: The composition (in %) of investigated Al-alloys.

The bending with rotation of cylindrical specimens of 12mm in diameter from the AVT-alloy was realized in the tension stress range of 100...150 MPa at frequency 10 Hz.

Specimens from D1T-alloy of 40x20 mm in their section were tested under uniaxial tension at the stress of 160 MPa at the stress ratio  $R=0.1$  and the frequency of 5 Hz. First, the semi-elliptically shaped crack was performed up to its sizes in the depth direction near to 10 mm and on the specimen surface near to  $2c=20$  mm. Then, the maximum stress level was only decreased down to the stress of 80 MPa and under this cyclic loads stress the crack length was increased up to the depth near to 20 mm.

Biaxial tension-compressive of the 10 mm thick cruciform specimens from AK6 Al-alloy (see Tab. 1) with semi-elliptical shaped cracks was performed under regular cyclic loads at frequency of 5 Hz and the principal stresses ratio  $\sigma_2 / \sigma_1 = \lambda = -0.9$  in the range of  $100 \text{ MPa} < \sigma_1 < 160 \text{ MPa}$  [16]. The biaxial stresses state was uniform within the central zone of the specimen of 20 mm in diameter.

Irregular cyclic loads with biaxial overloads were performed on the specimens from D16T Al-alloy (analogue of 2024T3) [17]. The spherical particles were formed on the fracture surfaces after the overloads factor  $Q_0$  of 1.8 and 1.5 when the shear lip width  $2t$  reached near to the half of the specimen thickness.

The fatigue surfaces were cut from specimens and their analysis was developed fractographically in the scanning electron microscope EVO40 of Karl Zeiss instruments with resolution 3 nm, CD50, and Hitachi.

Wear debris were seen on the fatigue surface between facets with fatigue striations pattern. Places with wear debris were oriented in the parallel direction to the crack growth. Three shapes of particles such as cylinder, ellipsoid, and sphere can be seen on the places with wear debris, Fig. 3.

The length of cylinders reached 70  $\mu\text{m}$  and they have diameter in the range of  $1...5 \mu\text{m}$ . Some particles are placed into oriented sockets. The cylindrical sockets are situated equidistantly one after another one. Their axes orientation being in the parallel to the crack growth direction is dominant. The cylindrical particles fragmentation have made as a result of their rotations between free surfaces. The spiral crack, as shown in Fig. 3, performs because of the twisting process of the particles fragmentation due to the mode III opening. The rotation created a specific border between fragments such-like as a conic-cavity on the one piece and a just out conic on the other one, Fig. 3. That confirmed the rolling mechanism of their formation from the fragments of the cylindrical particles. In some cases the particles associated with wear tracks, where they were removed by replicating tape. They apparently had greater hardness the matrix. The tracks orientation coincides with the crack surfaces moving due to mode III opening. The material heaped up from the tracks on the facets with fatigue striations pattern. Therefore this material heaped up from tracks sometimes later than the fatigue relief was formed.

After uniaxial overloads there is wear debris evident on the fracture surface following the stretched or dimpled zones at  $R=0.1$ . The density of debris decreases as the R-ratio increases. The original shapes of the wear debris were ellipsoidal and spherical particles formed on the fracture surface after overloads more than factor 1.5, Fig.4 (a).

As the double shear lip width increased near to the specimen's half-thickness (as the crack developed) the particles appeared the moment an overload was applied. They lay on facets, which are parallel the crack growth direction. Because



the spherical particles impeded crack opening, the crack growth is not so intensive after overloads, as was the case for regular loading at the same value of stress intensity factor when the shear lip development was very intensive.

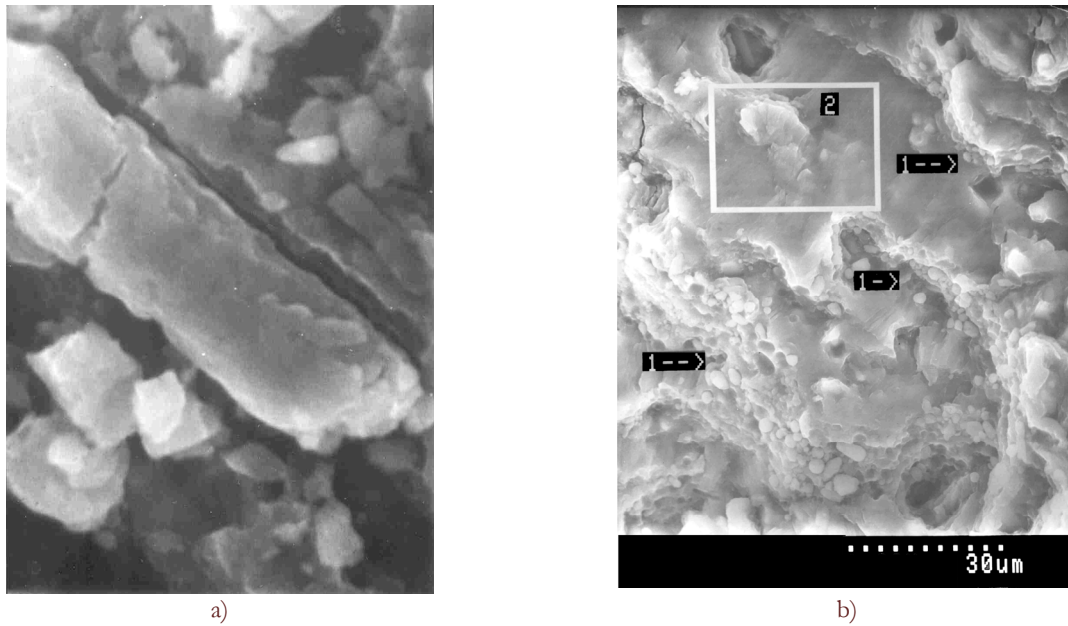


Figure 3: One of the cylindrical particles (a) with dislocation crack, and (b) the fracture surface (2) one of the mesotunnels with spherical particles (1) by the surfaces of collapsed crosspieces for a fatigued specimen of AK6 Al-based alloy tested under biaxial tension-compression.

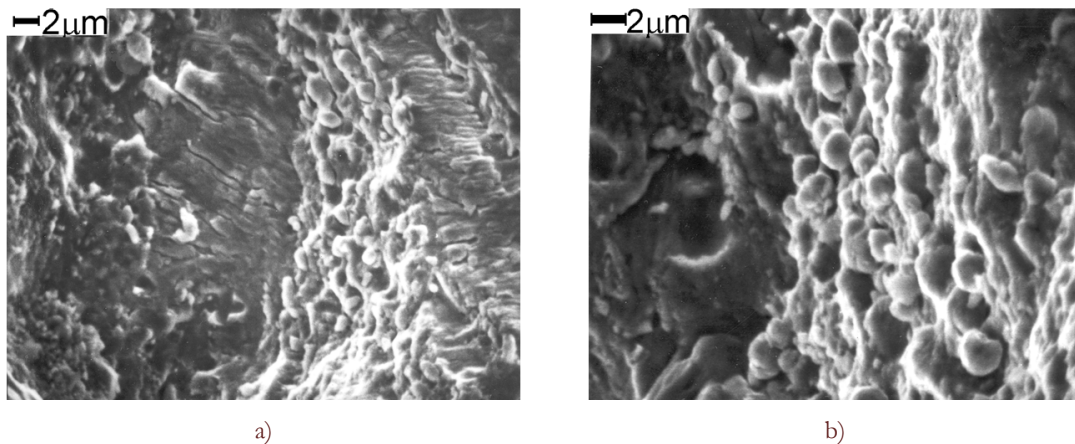


Figure 4: Spherical particles in area of cracked crosspieces of cruciform specimens of D16T aluminium alloy having formation during through crack propagation after single overloads with (a)  $Q_0 = 1.5$ ,  $\lambda_\sigma = 0$ ,  $\sigma_1 = 100$  MPa,  $R=0.5$ ; and (b)  $Q_0 = 1.5$ ,  $\lambda_\sigma = 0.4$ ,  $\sigma_1 = 130$  MPa,  $R=0.1$ .

Under biaxial overloads the fracture surface exhibits systematic behaviour. The wear debris in the form of particles appeared after the dimpled zone after overloads. During crack propagation they placed on the facet along the oriented along crack growth direction (see Fig.4 (b)) and their formation at regular loading develops under the compressive load acting in perpendicular direction of the crack growth direction. Overloads produce compresses stress ahead of a crack tip. It seems to be that the appearance of spherical particles indicates the development of the compressive load along the crack front.

Therefore the mechanism of spherical particles formation can be explained using the model of the permanent contact between fatigue surfaces in the mode III opening. The fatigue crack appearance and propagation through a metal realises

as a result of the mesotunneling effect. In the mode I of external cyclic loads crosspieces collapse can be seen by the shearing due to mode III opening. This is a twisting process.

The other way of the crosspiece collapse realises by the rotation instability effect shown in Fig.5. At the big plastic deformation process material volumes can be rotated into crosspieces. The cylindrical particles create, Fig. 3, (a), by borders of these volumes with cellular dislocation structures under the mode III crack opening. The cellular pattern could be seen on the particles surface before their rotations between free fracture surfaces. The crushed cylindrical particles under the twisting process due to mode III crack opening have a conic-cavity shape for border of one fragment and just out conic for another one. At last, the cylindrically-shaped particles become, first, ellipsoidal and, then, spherically-shaped particles because of rotations under mode III between free fracture surfaces under cyclic loads.

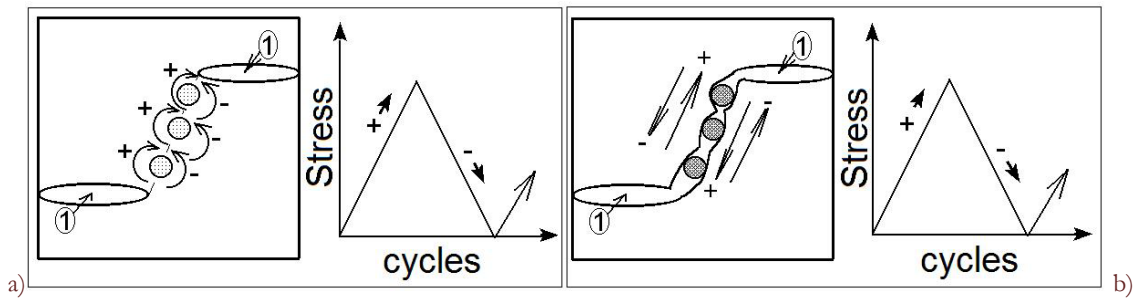


Figure 5: Schema of cascade events in fatigued metals during sequentially, first, cylindrical, then, ellipsoidal, and, at last, spherical particles formation because of shear and volume rotation in crosspieces between mesotunnels “1”.

So, the spherical particles formation process performs because of the mesotunneling effect, the rotation instability into crosspieces between tunnels, and free surfaces formation by the rotated volumes with cylindrically-shaped particles. Then, these particles crush, roll, and ellipsoidal-spherical particles form into crosspieces under mode III opening of mode I fatigue cracks growth.

#### OG'e spectroscopic analysis

The composition of wear debris with particles was analyzed on the fatigue surface of AVT alloy tested under the bending with rotation. The OG'e spectrometer LAS-2000 ("Riber", France), having the co-axial electronic gun, was used for the analysis. The receiver of electrons beam was such as "cylindrical mirror". The standard characteristics of the instrument are the next: the resolution -  $DE/E < 0.3\%$  at the rested pressure  $(1.3-2.6) \times 10^{-8}$  Pa, the current of the gun electron beam - near to  $5 \times 10^{-7}$  A, energy of the electron beam with diameter of somewhat microns was 3keV. The place for wear debris analysis on the fatigue surface in several quadratic millimetres was chosen in the regime of secondary electrons.

Reduction of fatigue surface layers by argon ions ( $Ar^+$ ), had having energy 3.5keV, was made in vacuum into OG'e spectrometer to analyze the profile of elements concentration for removed layers. First, the "Al" reduction rates near to 40 A/min have been demonstrated for such specimens as thin films of pure Al. The films' thickness was exactly known. This value was used to calculate elements concentration for fatigued specimens. The calculation was not exact because the fatigue surface was very rough but rates dispersion depended on the ions  $Ar^+$  angle downfall on various surface's places.

Interpretation of the OG'e electrons pike near to 55 eV was made on the basis of studies [18]. Its appearance is a result of Al oxidation up to  $Al_2O_3$ .

The local fatigue surface to be analyzed, was separated in two zones groups: 1- facets with particles (collapsed crosspieces); 2 - plates with fatigue striations pattern, formed in meso-tunnels. First, the spectrum of elements was demonstrated on the fatigue surfaces of both zones before surface reduction by ionic  $Ar^+$  in vacuum. The spectrum put together pikes of Al, O, S, P, Cl, K, Ca, N, C, and a big pike of oxygen. It was spectrum of surfaces dirty which had a good looking in one of the points specially chosen by their maximum of concentration, Fig. 6 (a). There was not principal difference in dirty patterns for zones 1 and 2 on the fracture surface.

After ionic  $Ar^+$  attack of surfaces in 15 minutes there was good looking pike of 68keV (Al) in zones 1, but pikes of elements from films of oxidized metal have disappeared, Fig. 6 (b). The pike of 99eV (Si) had a good looking, but pikes of P, S, Cl, K, C, Ca, N, and oxygen decreased. In 45 minutes of ionic  $Ar^+$  attack of the fracture surface the pikes of 68eV (Al) and 92eV (Si) had much better looking, but others decreased, Fig. 6 (c). The pike's amplitude for oxygen was unchangeable. At that time elements P-S-Cl-K-Ca were absolutely removed from zones 2. There were only small pikes of C-N with the good looking pike of the oxygen for these zones, Fig.6 (d). The aluminium intensity amplitude of 55eV had a good agreement with the intensiveness of the oxygen pike. The relationship between Al/O, calculated by the discovered

spectrum, was near to 2/3. Therefore the first pike of 55eV correlates with  $Al_2O_3$ , and the second pike of 68eV belongs to the aluminium.

Successive appearance and disappearance of various elements before and after ionic reduction of surface lyres in zones 1 correlated with the particles formation process on the fatigue surface which was described above. First, into the aluminium matrix the small particles of  $Al_2O_3$  were pressed. Second, under cyclic loads the film of the dirt covered that surface composition. The ionic surface lyres reduction has cleaned the composition from the dirty and the electron beam, having the diameter in the range of 1...4  $\mu m$ , has demonstrated the particles of  $Al_2O_3$  and Al-matrix.

Spectrums from the zones 1 and 2 were also analysed in another sequence of events. After the surface reduction by ionic  $Ar^+$  in 45 minutes the electron beam scanning was made in the approximately perpendicular direction to the crack growth direction. As a result, there was discovered the systematically maximum intensity of the C-pike from the surface in zones 1, but it had minimum in zones 2. There was not registered elements of the dirty in zones 1, but the metallic Al-68eV, many of the carbon and the oxygen were fixed, Fig. 6e. In zones 2 the dirt was registered, but the carbon pike was absent. The pikes of  $Al_2O_3$  and oxygen took place, Fig.6f. The rate of 68eV (Al) appearance shown that rates of ionic surface reduction for both zones were approximately the same.

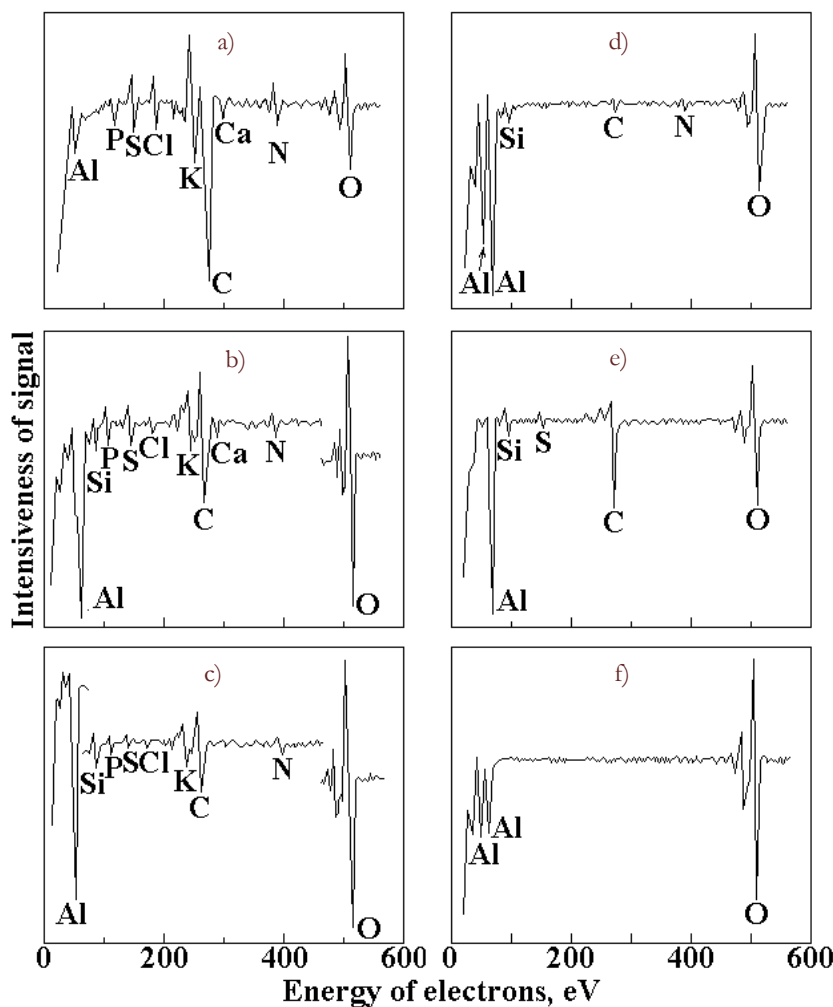


Figure 6: OG'e- spectres made from fracture surfaces of specimens of AVT Al-based alloy for (a) the initially performed fracture surface, after its removing under ions  $Ar^+$  during (b) 15 min, and 45 min for (c) zones-1, and (d) zones-2; (e) and (f) for zones-1 and zones-2 respectively but reproduced by the pike of carbon.

The local material heating under fatigue crack surfaces tearing made up oxidation by all surface including places with particles. The heating influenced carbonic films formation on fatigued crosspieces surfaces whose width is not less than 200 nm (0.2  $\mu m$ ). The process of the films formation performs as a result of carbonic-hydrogenous compositions



degradation around zones of material heating. Oils vapour compositions always exist in laboratory air around test machines. The others elements of S, Cl, P, N, K, Ca material also absorbs from the environment.

The pike of 55eV ( $\text{Al}_2\text{O}_3$ ) disappeared in 15 min under the fatigue surface ionic  $\text{Ar}^+$  reduction. This allowed to us to summarise results of particles investigations and to conclude that width of the  $\text{Al}_2\text{O}_3$  film for particles is not less than 60nm, Fig. 7.

The such elements as S, P, Ca and others are placed into the carbonic films because replacement of the pike of 68eV(Al) on the pike of 55eV ( $\text{Al}_2\text{O}_3$ ) performs quicker than pikes of the dirty elements disappear.

Results of performed investigations have shown that in the area of fretting damage of interacted parts of Al-based alloy there performed complicated processes, which directed to create not only oxides but nonuniform composition by the appeared surface

The small ellipsoidally-spherically-shaped particles of the oxidised aluminium, having sizes in the range of  $0.1...2 \mu\text{m}$ , are placed into the carbonic-dirty films. That is why the fretting wear debris from aluminium alloys have the black colour. This colour reflects the carbonic films formation, but it is not colour of the aluminium oxide.

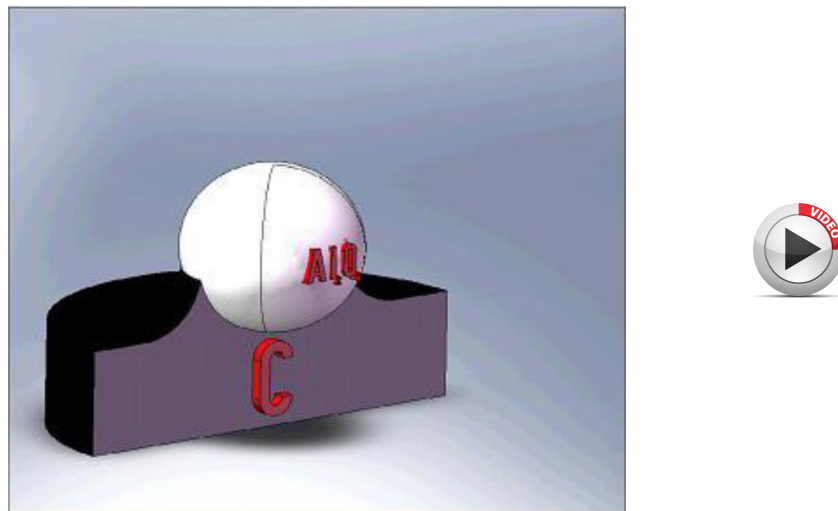


Figure 7: Schema one of spherical particles placed in “C” - carbon lyre, with indication of  $\text{Al}_2\text{O}_3$  as this particle skin.

## CONCLUSION

1. Fatigue fracture surfaces of Al-based alloys were performed under various cyclic loads. They were analysed in scanning electron microscope and such wear debris pattern as spherical particles were discovered in zones with the black colour. It was shown that spherical particles take only place on fatigue fracture surfaces for the Second stage of fatigue cracks development accompanying process of fatigue striation formation. Mesotunneling process is the first step of the material fatigue fracture along the crack front during fatigue crack propagation. Then crosspieces volume between mesotunnels had collapse experiencing simultaneously shear and rotation deformation.

2 The surfaces of collapsed crosspieces with spherical particles were investigated on the bases of the OG'e spectroscopic analysis and the sequence of events during the particles formation inside of the plastic zone and behind of a crack tip under cyclic loads were discovered. The elliptical or spherical shapes of wear debris build up on the fracture surface because of, first, rotation deformation processes in material volumes between mesotunnels, and second, crosspieces fracture with free surface formation by the border of rotated cylindrical volumes. Further, because of fretting process between crack edges by the surface of collapsed crosspieces, cylindrical particles transform in ellipsoidal and spherical particles with intensive oxidizing particles surface.

3. Nevertheless by the rotated volumes appeared films of black colure within which placed particles of different shapes. These films create because of local material heating under fatigue crack surfaces tearing and particles rotation. The heating influenced carbonic films formation on fatigued crosspieces surfaces whose width is not less than 200 nm ( $0.2 \mu\text{m}$ ). There oils vapour compositions always exist in laboratory air around test machines. The process of the films formation performs as a result of carbonic-hydrogenous compositions degradation around zones of material heating.





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