## Scanning Electron Microscopy

Volume 1986 Number 3 *Part III* 

Article 10

7-6-1986

# Scanning Electron Fractography of Body Centered Cubic (BCC) Metals

A. D. Vasilev Academy of Science of the Ukrainian SSR

Follow this and additional works at: https://digitalcommons.usu.edu/electron

Part of the Life Sciences Commons

## **Recommended Citation**

Vasilev, A. D. (1986) "Scanning Electron Fractography of Body Centered Cubic (BCC) Metals," *Scanning Electron Microscopy*: Vol. 1986 : No. 3 , Article 10. Available at: https://digitalcommons.usu.edu/electron/vol1986/iss3/10

This Article is brought to you for free and open access by the Western Dairy Center at DigitalCommons@USU. It has been accepted for inclusion in Scanning Electron Microscopy by an authorized administrator of DigitalCommons@USU. For more information, please contact digitalcommons@usu.edu.



### SCANNING ELECTRON FRACTOGRAPHY OF BODY CENTERED CUBIC (BCC) METALS

#### A.D. Vasilev

#### Institute for Problems of Materials Science Academy of Science of the Ukrainian SSR Kiev, 252180, USSR

(Received for publication June 10, 1985, and in revised form July 06, 1986)

#### Abstract

Scanning electron fractography is an inherent part of investigations of factors which determine the mechanical properties of materials and their failure. Transition bcc metals show the widest variety of fracture mechanisms under uniaxial tension.

Brittle fracture is affected by cleavage from various defects - stress concentrators.

In brittle-ductile transition, fracture starts by a "tough" mode but finishes by a brittle one - by cleavage. A fracture mechanism changes after the "tough" crack has reached some critical length. Mechanisms of subcritical growth are of cleavage with relaxation, intergranular fracture and dimpled ones.

Dimples are observed in ductile fracture. Cleavage is absent. The dimples are nucleated as a result of both failure of particles and their interfaces and delamination of structure elements.

All varieties of observed fracture surface may be described as a result of actions of the following mechanisms cleavage, cleavage with relaxation, pore coalescence, brittle intergranular or intercellular fracture.

Fractographical analysis allows one to obtain information not only about the fracture mechanisms but also such characteristics as: fracture toughness, brittle-ductile transition limits, structure transformations preceding fracture.

<u>KEY WORDS</u>: Bcc metals and alloys, Brittle-ductile transition, Uniaxial tension, Fracture, Fracture mechanisms, Fractography, Fracture toughness, Failure analysis

#### Introduction

Advent of modern scanning electron microscopes stimulated a new interest to fractography and caused a rapid growth of fractographic research. Scanning electron fractography has become an inherent part of investigations of factors which determine the mechanical properties and failure of materials. A number of reviews may be found in literature devoted to interpretation of fracture surfaces as well as mechanism of their formation /7,8,10, 18,26,27,41/ and conditions under which some of them are revealed in specific materials /1,19,21/.

Among fractographic studies of the last decade, experiments with transition bcc metals which are apt to brittle-ductile transition and show the widest variety of fracture mechanisms are of the most interest. They allow one to classify most fully various morphological types of fracture surfaces associated with these or those fracture mechanisms.

The given paper summarizes some fracture mechanism studies of materials on bcc metal base subjected to uniaxial tension. We shall also discuss other information which is contained in pictures of fracture surface, about such as energy conditions of failure, brittleductile transition limits, and so on.

	Fr	actur	e mec	hani	sms	
and	thei	r chai	nge a	at tr	ansit	tion
			from			
bri	ttle	state	into	duc	tile	one

Comparison of temperature dependences of mechanical properties of singlephase and particle-hardened materials on body centered cubic (bcc) metal base which have been found in references /2, 4, 9, 25, 31, 32, 46, 48, 53-55 and others/ and are shown schematically in Fig.1 with pictures of their fracture surfaces allows one to divide the temperature range under study into the following three regions: BRITTLE FRACTURE region, up to  $T_t^{\ell}$ ; BRITTLE-DUCTILE TRANSITION re-



Fig.1. Diagrams of temperature influence on mechanical properties and fracture mechanisms of single-phase (a) and particle-hardened (b) materials on bcc metal base under uniaxial tension: 1a,b - cleavage from pre-existing defects; 2a - cleavage from the Fridel - Orlov or intergranular cracks; 2b - cleavage from intergranular cracks; 3a - cleavage from dimpled cracks; 4b - dimpled localized fracture; 4a,5a - dimpled fracture.  $\Im_Y$  - yield strength,  $\Im_{\mathcal{F}}$  - fracture strength,  $\mathscr{O}$  - elongation,  $\mathscr{Y}$  - narrowing.  $\mathcal{T}_{\mathcal{L}}^{\ell}$  and  $\mathcal{T}_{\mathcal{L}}^{\mu}$  are the lower and the upper limits of brittle-ductile transition, respectively.  $\mathcal{T}_{\mathcal{C}}$  is temperature at which a mechanism of subcritical crack growth is changed.  $\mathcal{T}_{\mathcal{L}}^{\mu'}$  is temperature at which size of dimples is abruptly changed in particle-hardened materials.

gion,  $T_t^{\ell} - T_t^{\mu}$ , which, in its turn, may be divided into two subregions,  $T_t^{\ell} - T_c$  and  $T_c - T_t^{\mu}$ , and DUCTILE FRACTURE region, above  $T_t^{\mu}$ , where two subregions may be distinguished in particle-hardened materials:  $T_t^{\mu} - T_t^{\mu'}$  and above  $T_t^{\mu'}$ . Width of these regions and subregi-

Width of these regions and subregions, as well as their limits, depend on material and especially on its structure. The same mechanisms, which are observed in some materials, e.g., intergranular fracture in a number of Mo alloys /50,54/ in an extremely wide (several hundred degrees) temperature range, manifest themselves in other materials in a very narrow (hardly some dozens of degrees) temperature range, and, hence, the very existence of these mechanisms becomes problematic, e.g., intergranular fracture in V alloy /5/. Only does consideration of all scales of material (single crystals, single-phase and particle-hardened alloys) allow one to construct the general scheme of modification of fracture mechanisms at transition of materials on bcc metal base from the brittle state into the ductile one.

Consider now the fractographical peculiarities of fracture in the temperature regions stated above.

Brittle fracture. In the region of brittle fracture, at temperatures below  $T_t^I$ , samples fail by cleavage at stresses below their flow stresses. Fracture is not preceded by noticeable plastic strain (fig.1). Fractographical analysis unambiguously shows that the cleavage crack originates from inner or outer pre-existing defects of the sample which satisfy the Griffith condition /24/. Bcc metals cleave usually along

planes where the surface energy is minimum, i.e., along planes of the {100} family, although cleavage along other planes, e.g., {110}, may be observed. Formation of the cleavage surface

has been treated in detail by Gilman /23/, Low /29/, Beachem /7,8/ and other authors. Therefore, we shall discuss here only such important elements of cleavage as "the step" and "the river pattern".

"The step" results from a joining of cleavage cracks produced when a crack has passed, e.g., a twist boundary or a screw dislocation (fig.2a). New cleavage cracks do not appear while crossing a tilt boundary or an edge dislocation. In this case, only tilt of crack is changed (fig.2b).

Joining of a large number of cleavage cracks or, using a generally accepted term, confluence of cleavage steps forms "the river pattern" (fig.2a). The main value of the river pattern is that it allows one to determine the direction of crack propagation: a crack has run in direction of confluence of cleavage steps. By the way, the river pattern may be, in principle, found in ductile fracture too. In this case, the pattern is named "chevron" /10/.

Morphology of cleavage in materials containing particles has some peculiarities connected with both morphology of







Fig.2. Transition of cleavage through a) a twist boundary with formation of steps and river pattern and b) a tilt boundary. Cr alloy at  $T=20^{\circ}C$ .

particles themselves and value of their bonds with matrix. In materials containing plain particles, the cleavage surface fragmentation is observed (fig.3a)/2, 4,5/. Size of the fragments depends on dimension of particles and distance between them. When a cleavage crack passes through a particle strongly bound to matrix, the relief does not reveal marked changes. But when the bond is weak particles are separated from the matrix and cleavage transfers to another plane (fig.3b). This implies that crack propagation is retarded by particles in accordance with Cook and Gordon /15/.

<u>Brittle-ductile transition</u>. Fracture in temperature range of brittle-ductile transition  $(\mathcal{T}_t^\ell - \mathcal{T}_t^\mu)$  occurs after some plastic strain (fig.1) and is characterized by the fact that its start and development to a definite limit are



Fig.3. Pictures of cleavage in particlehardened alloys: a) fragmentated cleavage in V alloy at T=-4°C; b) transition of cleavage through particles weakly bound with matrix in Mo alloy at T=-196°C. Delaminations of particles from matrix and transfer of cleavage to another plane may be seen.

realized by one mechanism but is completed by another one in a brittle mode by cleavage. Fractographical analysis of the cleavage river pattern gives us opportunity to distinguish these two fracture stages: a stage of "tough" subcritical growth of a crack and a stage of its brittle catastrophic propagation (fig. 4a, b). Change of fracture mechanisms and the very transition from tough fracture to brittle one occur after the "tough" crack has attained some critical length /48/ according to the Griffith condition /24/.

Critical size of a tough part of the crack and the mechanism of its growth depend on temperature /52/ and



gle-phase Mo materials at T=20°C: 1 - a stage of subcritical growth, 2 - a stage of catastrophic cleavage. a) single crystals of Mo [100],

- b) polycrystalline Mo alloy,c) striations of subcritical stage. Higher magnification of b) crack.

rate of loading /51/. According to the growth mechanism of subcritical cracks all temperature range of brittle-ductile transition is subdivided into two subre-gions:  $T_t^{\ell} - T_c$  and  $T_c - T_t^{\mu}$  (fig.1). At  $T_c$  temperature, a growth mechanism is changed, and a neck is formed in sample. In the first transition subregion  $(T_t^{\epsilon} - T_c)$ , fracture occurs after relatively small plastic strain. A mechanism by which the crack grows to critical size depends on structure. In single crystals and single-phase polycrystals which are not apt to intergranular fracture, the crack grows by jump-like overcoming of its inherent zone of plastic relaxation. Specific features of such crack are striations (fig.4c) and apparent likeness to fatigue crack (see, e.g., /10/). Relief of these cracks depends on temperature and crystellographic orientation. Such cracks were observed by Hull et al on Mo, W and Fe-Si notched single crystals /9,25,37/, in LiF crystals by Gilman et al /22/ and in ceramics by Stocks /42/. The striated relief may be result from comparatively slow propagation of cleavage crack with periodic relaxation. Going from Gilman works /22 and others/ Fridel /20/ and independently Orlov /35/ gave an appro-ximate scheme of this process. In this connection, we shall name the cleavage with relaxation as the Fridel-Orlov mechanism.

In materials with weak grain boun-daries cleavage is preceded by intergranular fracture which may be both brittle (fig.5a) and ductile by pore coalescence (fig.5b). Intergranular cracks are generated in process of plastic deformation by sliding /38/ or twinning that has preceded fracture. Traces of this deformation may be observed on the grain bo-undaries as steps of slipping (fig.6a) or twinning (fig.6b) shifts. Considered above intergranular cracks have approximately transverse orientation to tensile stress.

When temperature rises to  $\mathcal{T}_{c}$  intergranular cracks of another type appear in a fracture surface. They orient themselves along exis of a sample and delaminate it (fig.7). Beginning from  $T_c$ temperature, this process is developed to such extent that transverse intergranular cracks are not observed. The sample is divided by the delaminating cracks into microsamples - everyone of which is fractured by itself either plastically with the formation of knife-like fracture or in brittle mode by cleavage. In addition, the misoriented dislocation cell structure is formed in neck of sample. It leads to appearance of intercellular delaminating cracks. Quantity of such cracks also increases with a rise of testing temperature /54/.







Fig.5. Pictures of intergranular fracture of particle-hardened alloys: a) brittle one in Mo alloy at T=210°C, b) ductile one in Nb alloy at T=20°C.

In materials which are deformed via twinning and have strong grain boundaries, e.g., in silicon iron, the cleavage begins from cracks produced by fracture of twins (fig.8a) or from intertwin cracks (fig.8b).

At temperature above  $T_c$ , fracture is preceded by significant plastic deformation (fig.1). The neck is developed in the sample. Subcritical cracks grow by pore coalescence (fig.9a,b). When the sample flow is suppressed by a notch, the upper temperature limit

When the sample flow is suppressed by a notch, the upper temperature limit of the Fridel-Orlov mechanism range is shifted to the higher temperatures, practically up to  $T_{t}^{\mu}$ . In this case, intergranular fracture may be not observed (excluding case where a grain boundary lays in a plane of the Fridel-Orlov crack). Hence, it may be concluded that the Fridel-Orlov mechanism is valid only



Fig.6. Traces of plastic deformation on surfaces of intergranular fracture in Fe alloy: a) traces of slipping at  $T=-100^{\circ}C$ , b) traces of twinning at  $T=-196^{\circ}C$ .



Fig.7. Delaminating crack and cleavage in Mo alloy at T=200°C.





Fig.8. Cleavage from intratwin cracks in Fe-Si alloy at  $T=-80^{\circ}C$  (a) and surface of intertwin crack in Armco-Fe at  $T=-196^{\circ}C$  (b).

for the plain strain, when deformation constraint takes place but general sample flow promotes to develop intergranular fracture and fracture by pore coalescence. It also follows from this that under conditions where general flow is impossible the Fridel-Orlov mechanism is the only mechanism of subcritical crack growth even in meterials with sufficiently weak grain boundaries.

Cleavage observed in temperature range of brittle-ductile transition at the stage of fracture completion is characterized by:

1. Tear ridges. They are due to the higher toughness and plasticity of material and result from ductile fracture of bridges between cleavage cracks under shear stresses. In particle-hardened alloys the bridges are observed to be fractured by pore coalescence (fig.10a).





Fig.9. Two stages of crack growth in particle-hardened alloys. 1 - a stage of subcritical growth, 2 - a stage of catastrophic cleavage. a) Cr alloy at  $T=200^{\circ}C$ , b) V alloy at  $T=20^{\circ}C$ .

The cleavage with ridges is often called "quasicleavage", "tough cleavage", "cleavage III" /1,21/. 2. More rough relief. It is caused

2. More rough relief. It is caused by new structure formed by a plastic deformation preceding fracture. Slide bands and dislocation cells are displayed as the strips (fig.10b). Twins are seen as "tongues" (fig.11a), X-shaped (fig.11b) and other pictures. It is possible to observe ductile fracture of twins with formation of knife-like (fig. 11c) or dimpled (fig.11d) fracture.

Ductile fracture. At temperatures above  $\overline{T_{t}^{\mu}}$ , ductile fracture takes place. Samples fail via the nucleation, growth and coalescence of pores\*). Fracture is

\*) In the western literature the term "void", but not "pore", is usually used. The sample becomes porous, not empty, so, the term "pore" is more acceptable.

preceded by significant deformation (fig.1). Fig.12a shows a typical picture of ductile fracture. Dimples are observed. In single crystals the so-called knife-like fracture is often formed (fig.12b).

The most important stage of ductile fracture process is nucleation of pores.

It is usually assumed / 16,17,28,30, 43 and others/ that pores nucleate due to presence of particles. This is supported by the fact that, as Cryssard et al./16/ have shown, particles or their fragments are found in dimples. Really, in materials where particles are weakly bound to matrix, pores may be formed already at a stage of elastic strain through the Olsen-Ansell mechanism /34/. In materials where the interface is strong, pores are created by plastic deformation according to Broek / 11-13/.

However, dimpled fracture is observed not only in materials containing particles but also in single-phase materials and even in single crystals (figs. 12c,d). It was repeatedly noted /6,14, 40/ that there is no simple correlation between quantity of dimples and quantity of particles. Bauer and Wilsdorf /6/ showed that density of dimples exceeds density of particles by a factor of about two. It evidently shows that the particles are not necessary for the pore nucleation.

Investigation of effect of dislocation structure on a fracture surface of materials free from particles has shown that with growth of plastic deformation before fracture, and with growth of perfection and misorientation of dislocation cellular structure, fracture varies from cleavage to intergranular, intercellular one, more close by it's shape to dimpled fracture. Distance between large dimples corresponds to grain size while distance between small dimples corresponds to cell size. It has been suggested that ductile crack propagates by successive rupture of structure elements (grains, cells) as microsamples with fo-rmation of the knife-like fracture in every microsample. Pores are nucleated on boundaries of structure elements due to delamination /49/. This conclusion has been proved by Wilsdorf et al /36, 57/ when they deformed their thin foil samples in a high voltage electron microscope.

The pores may also result from formation of disclination /39,56/ or interaction of twins between themselves and grain boundaries /47/. Large pores-pipes originate mainly at a triple joint of grains /53/.



Fig.10. Peculiarities of fracture by cleavage on a stage of catastrophic crack propagation: a) fracture of bridges between cracks by pore coalescence ("dimpled cleavage steps") in V alloy at T=200°C, b) striated contrast due to slip band in single crystal of Mo [100] at T=200°C.

In materials with intense delemination, e.g., in Mo and W, temperature interval may be pointed where fracture varies from delaminated mode to dimpled one /31,32,48,55/.

Study of particle-hardened alloys has shown that in temperature range  $T_t^{u}$ - $T_t^{u}$  ductile fracture occurs after relatively small deformation (fig.1b) under its localization in a thin layer of sample. Crack runs in a deformation band /33/. Fracture surface is characterized by small dimples elongated in direction of crack growth. Particles are observed in the dimples (13a).

At temperatures above  $\mathcal{T}_{t}^{\mu\prime}$ , plastic deformation before fracture increases







Fig.11. Pictures of fracture of twins in Fe-Si alloy at  $T=-80^{\circ}C$  (a,b) and Armco-Fe at  $T=-196^{\circ}C$  (c,d): a) twin tongues; b) X-shaped fracture; c) knife-like fracture; d) dimpled fracture.

sharply (fig. 1b); size, shape and orientation of dimples change. The dimples are large now, deep and elongated in direction of normal stress. There are no particles in the dimples (fig.13b). It has been found from a longitudinal cleahas been found from a fongitudinal clea-vage of fractured samples that at tempe-ratures below  $T_t^{\mu\prime}$  there are cracks and pores around the particles (fig.14a), while above  $T_t^{\mu\prime}$  they are absent (fig.14b). This shows that above  $T_t^{\mu\prime}$  particles are not more efficient obstacles for dislocations. Dislocations bypass the particles by transverse sliding. Particlehardened alloys behave as single-phase ones.

### Fracture mechanisms in bcc metals

As follows from the above stated, all varieties of observed fracture surfaces may be described as a result of

action of such mechanisms or their combinations as:

- cleavage,
- the Fridel-Orlov mechanism.
- pore coalescence, brittle intergranular or intercellular fracture.

Intragranular fracture occurs by cleavage, the Fridel-Orlov mechanism or pore coalescence, while intergranular fracture occurs either in brittle mode without or with traces of plastic defor-mation, or in ductile one by pore coalescence.

#### Other information contained in fracture surface

The main information drawn out from the fracture surface is undoubtedly information about mechanisms and origins of fracture. At the same time, as it fol-









Fig.12. Pictures of ductile fracture: a) dimples in Fe alloy caused first of all by nonmetallic inclusions at T=20°C; b) knife-like fracture of Mo [110]at T=-80°C; c) dimples in Mo [100] at T=600°C; d) the same, at T=800°C.

lows from the above presented results, fracture surface may give other not less important information, e.g., about such properties and characteristics of material as:

<u>Fracture toughness.</u> In temperature range of brittle-ductile transition two stages of crack propagation are distinguished: stage of a "tough" growth of the crack and stage of its brittle catastrophic propagation. Assuming that critical conditions, acceptable in fracture mechanics, are realized in a sample at moment of change of fracture mechanism, it is possible to determine some criteria of fracture mechanics, such as effective surface energy  $\mathcal{J}_{ef}$  or stress intensity factor  $K_{cc}$ , because we know the length of subcritical crack and fracture stress.

It has been shown /51,52/ that fracture toughness is controlled by the same thermoactivated processes that as the flow stress, and dependences of effective surface energy on temperature  ${\cal T}$  and rate  $\dot{\cal E}$  of loading may be described by relations

 $\mathcal{F}_{ef} = \mathcal{F}_o + Aexp\{-2\mathcal{U}_o/3kT\}$ , at  $\dot{\varepsilon} = \text{const}$ , (1)

 $\mathcal{F}_{ef} = \mathcal{F}_{o} + B/(\dot{\epsilon})^{2/3}$ , at T = const, (2)

where  $\mathcal{K}$  - true surface energy,  $\mathcal{U}_{o}$  - activation energy of dislocation motion, k - the Boltzmann constant, A, B -constants.

Temperature limits of brittle-ductile transition. At some temperature  $T_t^e$ , fractographical analysis allows one to find out the subcritical stage of fracture and to show that just a crack produced at this stage has served as a "defect" which has caused the subsequent catastrophic failure by cleavage (figs. 4,9). The  $T_t^e$  temperature also corresponds to point where curves of tempera-







Fig.13. Pictures of dimpled fracture of particle-hardened Mo alloy at a) T=420 °C and b) T=600 °C.

ture dependences of the fracture strength and the yield strength cross (fig.1). At the same time at  $\mathcal{T}_t^{t}$ , the material plastic characteristics differ from zero and start to increase. These three facts (appearance of the subcritical cracks,  $\mathfrak{S}_f(\mathcal{T}) = \mathfrak{S}_g(\mathcal{T})$  and  $\Psi, \delta > O$ ) permit one to define the  $\mathcal{T}_t^{t}$  as the lower temperature limit of brittle-ductile transition.

At  $\mathcal{T}_t^u$  temperature, fracture by cleavage is no longer observed. It allows one to define the  $\mathcal{T}_t^u$  temperature as the upper limit of brittle-ductile transition. Curves of mechanical characteristics vs temperature (fig.1) do not show any observable changes which could make more exact the value of this temperature. Hence, the determination of the upper limit of brittle-ductile transition as temperature at which any signs of cleavage disappear from fracture is a single



Fig.14. Interaction of particles with matrix in Mo alloy at different temperatures revealed by additional cleavages at  $T=-196^{\circ}C$ : a) cracks around particles due to their delamination from matrix at  $T=420^{\circ}C$ , b) cracks around particles are not seen. Particles were not delaminated from matrix at  $T=600^{\circ}C$ .

one at present. Above determination is consistent with the well-known determination of the transition temperature limits from a share of fibrous fracture at visual study of impact samples.

Besides, for particle-hardened alloys in region of ductile fracture, temperature  $\mathcal{T}_{\ell}^{\mu'}$  exists at which dimple size grows abruptly. The same is observed in austenitic stainless steel where small dimples become large ones when testing temperature varies from -196°C to room temperature /10/. At  $\mathcal{T}_{\ell}^{\mu'}$ , plasticity before fracture increases abruptly (fig.1b) and then particle-hardened materials behave as single-phase ones.

Grain boundary state. Some polycrystalline materials are apt to intergranular fracture under certain conditions. The fact that it is impossible to obtain even a few intergranular cracks suggests an extremely high strength of intergranular binding. Opening of boundaries by intergranular fracture provides us opportunity to analyse their chemical composition by Auger or X-ray microanalysis and crystallographic orientation by electron channeling pattern method. Usually analysis of grain boundaries is practically impossible when metallographical samples or thin films are used.

Surface of intergranular fracture also gives information about particles, their morphology and distribution (fig. 5). Brittle film precipitations are destroyed and fallen away from the grain body. Ductile streaks along the boundaries suffer ductile fracture and reveal dimpled intergranular fracture (fig.5b). At nonuniform distribution of intergranular precipitations brittle and ductile intergranular fracture may be simultaneously observed /2/.

Presence of particles. It is usually not difficult to detect particles weakly bound to matrix and having rather large sizes. But, as a rule, small particles are not seen in fracture. They may be indirectly discovered by investigation of fragmentation of the cleavage surface (fig.3a). Size of separate fragments gives information about size of particles as well as distance between them in dependence on particle orientation /5/.

Adhesive strength of particles with matrix. It is very important to determine the adhesive strength between particles and matrix, even qualitatively. One of the ways to solve this problem is to establish the SD-effect /3,34,44/, but the same qualitative estimation of the adhesive strength of particles with matrix may be also obtained from fractographical studies.

In cases, when the binding is weak, crack may be observed on matrix-phase interfaces (fig.3b), while at the strong binding they are absent (fig.14b).

Particles of a plate shape cause the fragmentation of cleavage surface (fig.3a). Absence of such fragmentation, e.g., in steels, says about the strong binding between cementite particles and matrix, while the fact of fragmentation seems to imply some average level of the binding strength.

Strength of particle-matrix interface may also be estimated from ductile fracture. If the binding is not strong, particles are found in dimples (figs.5b, 12a,13a), but there are no particles at strong binding (fig.13b). <u>Structure changes preceding fractu-</u> <u>re.</u> There are the following signs of changes that take place in structure before fracture:

- the striated contrast of cleavage surface (fig.10b). It indicates preceding deformation by sliding or twinning. Sliding gives more regular picture. Twinning gives rare strips.

- the intercellular fracture. It indicates formation of misoriented cellular dislocation structure.

- the twin pictures. Large figures are referred to deformation preceding crack formation. Small figures are produced just a moment of crack passage.

#### Use of additional cleavage

To get additional information about material under test a longitudinal section of fractured sample by the cleavage should be prepared. This technique is usually adopted to establish relations between structure and crack, but it can also be used to solve the following problems:

- effect of particles on deformation process. E.g., Fig. 14a shows pores around the particles in cross-section by cleavage and there are the particles in dimples of usual fracture too (fig. 13a). It shows that such particles have served as obstacles for dislocations at temperature 410°C /45/. But, at the same time, these particles have been not efficient obstacles for dislocations at 600°C. The dislocations do not pile near particles and do not break their interfaces. There are no cracks along these boundaries (fig.14b). The particles in dimples of fracture are also absent (fig.13b).

- disintegration of material into microsamples by delamination along grain boundaries (fig.15a).

- morphology of pores and places of their origins at ductile fracture (figs. 15 b-d).

- dynamic recrystallization (fig. 15d).

#### Conclusions

1. All varieties of fracture surfaces may be described as combination of a limited number of elements produced under action the following mechanisms: cleavage, the Fridel-Orlov mechanism, intergranular and intercellular fracture, pore coalescence.

2. Rise of temperature and, hence, growth of metal plasticity results in more complex cleavage relief in the following succession: smooth, "mirror" cleavage, practically with no relaxation; cleavage with steps; cleavage with periodic relaxation (the Fridel-Orlov mecha-



Fig.15. Preceding fracture transformations revealed by cleavages of fractured samples at T=-196°C: a) disintegration of Mo alloy sample into microsamples resulted from delamination along grain boundaries at T=400°C; b,c) pores on grain boundaries in Mo alloy at T=600°C and in Fe alloy at T=20°C, respectively; d) long pore-pipe and brittle intergranular fracture owing to dynamic recrystallization of Mo alloy at T=1000°C.

nism) and cleavage with ridges. Rise of temperature results in growth of dimples of ductile fracture, change of their morphology and orientation.

3. Depending on material purity and testing temperature intergranular fracture may be: brittle, almost with no traces of plastic deformation; brittle with traces of plastic deformation and ductile by pore coalescence.

4. Pores whose coalescence produces ductile fracture, in material free from particles, originate along boundaries of structure elements, but in materials with particles they result from failure of particles and their interfaces.

5. In temperature range of brittle-

ductile transition, fractographical analysis allows one to distinguish stages of the subcritical slow crack growth and the catastrophic crack propagation by cleavage. Mechanisms of the subcritical growth are as follows: the Fridel-Orlov mechanism, intergranular fracture and pore coalescence.

6. Analysis of fracture surface allows one to obtain information not only about origins and mechanisms of fracture but also about such characteristics of materials as: fracture toughness, limits of brittle-ductile transition, presence of secondary phase precipitates and strength of their bonds with matrix and structural transformations preceding fracture.

#### References

1. Ashby MF, Gandhi C, Taplin DMR. (1979). Fracture-mechnism maps and their construction for f.c.c metals and alloys. Acta Met. 27, 699-729.

2. Bankovsky OI, Vasilev AD, Gordeeva TI, Slepchishin LV, Moiseev VF, Trefilov VI. (1976). Fracture of particle-hardened vanadium alloy after various conditions of ageing. Ukranian Phys. J. <u>21</u>, 813-818, in Russian.

3. Bankovsky OI, Barabash OM, Vasilev AD, Moiseev VF. (1977). Dependence of yield strength of particle-hardened alloys from loading scheme. Powder Metallurgy, <u>5</u>, 50-56, in Russian.

4. Bankovsky OI, Vasilev AD, Malashenko IS, Moiseev VF, Trefilov VI. (1977). Fracture mechanisms of dispersion strengthened cast niobium alloy. Problems of Strength. 7, 88-94, in Russian.

5. Bankovsky OI, Vasilev AD, Malashenko IS, Moiseev VF, Trefilov VI. Fracture of particle-hardened vanadium alloy. Problems of strength.  $\underline{3}$ , 102-110, in Russian.

6. Bauer RW, Wilsdorf HGF. (1973). Void initiation in ductile fracture. Scr. Met. 7, 1213-1220.

7. Beachem CD, Pelly RMN. (1968). The electron fractography is a mean of fracture micromechanism investigation, in: Applied fracture toughness. Mir, Moscow, 311-346, in Russian.

8. Beachem CD. (1973). Microprocesses of fracture, in: Fracture, H. Liebovitz (ed). vol. 1, Mir, 265-375, in Russian.

9. Beardmore P, Hull D. (1967). Crack propagation in single crystals of molybdenum, in: Refract. Met. and Alloys, IV. Res.and Develop. vol 1. Gordon & Breach Sci. Publ. NY, 81-94.

10. Boyer HE. (ed.) (1984). Fractography and Atlas of Fractographs. Metals Handbook Vol. 9. ASM, Metals Park, OH 44073, 489p.

11. Broek D. (1971). A study on ductile fracture. Report NRC-TR-71021U, National Aerospace Lab. The Netherlands, Delft, 30-57.

12. Broek D. (1973). The role of inclusions in ductile fracture and fracture toughness. Eng. Fract. Mech. 5, 55-66.

13. Broek D. (1974). Some contributions of electron fractography to the theory of fracture. Intern. Met. Rev. 19, 153-182.

14. Burghard HC. (1974). The influence of precipitate morphology on microvoid growth and coalescence in tensile fracture. Met. Trans. <u>5</u>, 2083-2093.

15. Cook J, Gordon JN. (1964). On distribution stresses around top of moving crack. Proc. Roy. Soc. <u>A282</u>, 508-511.

16. Cryssard S, Plato J, Tamkhancar R, Anri G, Lageness D. (1963). Comparison of ductile and fatigue fracture, in: Atomic mechanism of fracture, Metallurgizdat, Moscow, 535-574, in Russian.

17. Embury JD. (1983). Ductile fracture, in: Strength of metals and alloys (ICSMA6), Proc. 6th Int. Conf. Melbourne, 16-20 Aug. 1982, vol. 3, 1089-1103.

18. Erasmus LA. (1974). Fracture modes in metals. New Zealand Eng. <u>29</u>, 219-226.

19. Fields RI, Weerasooriya T, Ashby MF. (1980). Fracture-mechanism in pure iron, two austenitic steels and one ferritic steel. Met. Trans. <u>All</u>, 333-347.

20. Fridel G. (1963). Strain hardening and crack propagation, in: Atomic mechanism of fracture, Metallugizdat, Moscow, 504-534, in Russian.

21. Gandhi D, Ashby MF. (1979). Fracture mechanism maps for materials which cleave: f.c.c., b.c.c. and h.c.p. metals and ceramics. Acta Met. <u>27</u>, 1565-1602.

22. Gilman JJ, Knudsen C, Welsh WR. (1958). Cleavage cracks and dislocations in LiF crystals. J. Appl. Phys. <u>29</u>, 601-607.

23. Cilman JJ. (1963). Cleavage, ductility and toughness of crystals, in: Atomic mechanism of fracture. Metallurgizdat, Moscow, 220-253, in Russian.

24. Griffith AA. (1920). The phenomenon of rupture and flow in solids. Phil. Trans. Roy. Soc. <u>A221</u>, 163-198.

25. Hull D, Beardmore P, Valintine AP. (1965). Crack propagation in single crystals of tungsten. Phil. Mag. <u>12</u>, 1021-1041.

26. Karel W. (1976). Introduction to the electron fractography of steel. Fraiberg investigation issue, <u>B172</u>, Fractography, 3-19, in German.

27. Kurdiumova GG, Milman JV, Trefilov VI. (1979). On the fracture mechanisms classification. Met. Phys. <u>1</u>, 55-62, in Russian.

28. Le Roy G, Embury JD, Edward C, Ashby MF. (1981). A model of ductile fracture based on the nucleation and growth of voids. Acta Met. 29, 1509-1522.

29. Low JR. (1963). Review of microstructure peculiarities at fracture by cleavage, in: Atomic mechanism of fracture, Metallurgizdat, Moscow, 84-108, in Russian.

30. Low JR. (1974). Observations of cavity nucleation, growth and coalescence in dimpled rupture, in: Proc. Intern. Conf. "Prospects of fracture mechanics," Delft Univ. The Netherlands, 24-28 June. Nordhoff International Publishing, Leyden, 35-49.

31. Morgunova NN, Kozakova NI. (1979). Mechanical properties and fracture mode of Mo alloys in temperature region of brittle-ductile transition. Met. Sci. and Heat Treat. <u>5</u>, 29-33, in Russian.

32. Morgunova NN, Solovjova NA. (1979). Fracture surface pecularities of deformed Mo alloys. Met. Sci. and Heat Treat.  $\overline{7}$ , 28-29, in Russian.

33. Niwa N. (1978). Shear bands in the "Cup and Cone" fracture process of pure titanium plate. J. Jap. Inst. Met. <u>42</u>, 1060-1066.

34. Olsen RJ, Ansell GS. (1969). The strength differential in two-phase alloys. Trans. ASM, <u>62</u>, 711-720.

35. Orlov AN. (1961). Long-term strength and stationary creep of polycrystals. Sol. St. Phys. 3, 500-504, in Russian.

36. Pellock TC, Wilsdorf HGF. (1983). Beryllium fracture observed by in situ high voltage electron microscopy. Mater. Sci. and Eng. 61, 7-15.

37. Pilkington R, Hull D. (1973). Sharp crack propagation in Fe-3, 5% Si single crystals, in: Fracture toughness of high strength materials. Metallurgia, Moscow, 9-19, in Russian.

38. Ribin VV. (1973). Intergranular fracture mechanisms in nickel. Phys. Met. and Met. Sci. <u>35</u> 993-998, in Russian.

39. Ribin VV, Vergasov AN, Likhachov VA, (1974). Ductile fracture as consequence of structure frag mentation. Phys. Met. and Met. Sci. <u>40</u>, 620-624, in Russian.

40. Romaniv VV. (1974). Electron fractography of strengthened steels. Naukova Dumka, Kiev, 98-102, in Ukrainian. 41. Schmitt-Thomas K, Klingele H, Woitcheck A. (1970). The micromorphology of metallic fracture. Pract. Metallography, <u>10</u>, 538-560.

42. Stocks PJ. (1976). Fracture microprocesses in ceramics, in: Fracture, H. Liebovitz (ed). vol. 7a. Mir, Moscow, 129-220, in Russian.

43. Thompson AW, Weinrauch PF. (1976). Ductile fracture: nucleation at inclusions. Scr. Met. <u>10</u>, 205-210.

44. Trefilov VI, Moiseev VF, Pechkovsky EP, Barabash OM, Bankovsky OI. (1977). SD-effect in particle-hardened Mo. alloys. Ac. Sci. Ukrainian SSR Reports, A1, 601-607, in Russian.

45. Trefilov VI, Pokhodnia IK, Vasilev AD, Malashenko IS, Moiseev VF, Pechkovsky EP. (1977). Fracture of molybdenum alloy with 3.5 vol.% of TiN. Problems of Strength, 5, 27-31, in Russian.

46. Trefilov VI, Pokhodnia IK, Moiseev VF, Vasilev AD. (1980). Ductile-brittle transition in refractory metal alloys containing dispersed second phase. Phys. Stat. Sol. (a), <u>59</u>, 843-851.

47. Van Stone RH, Cox TB, Low Jr. JR, Psioda JA. (1985). Microstructural aspects of fracture by dimpled rupture. Int. Met. Rev.  $\underline{30}$  (4), 157-179.

48. Vasilev AD, Malashenko IS, Pisarenko VA, Postnow AM, Trefilov VI, Firstov SA. (1977). Fractographical peculiarities of fracture of polycrystalline molybdenum at transition from brittle fracture to ductile one. Problems of Strength, <u>4</u>, 91-99, in Russian.

49.Vasilev AD, Malashenko IS, Trefilov VI, Shigonova OP, Firstov SA. (1977). Influence of plastic strain on structure and fracture mode of molybdenum. Phys. Met. and Met. Sci. <u>43</u> 640-644, in Russian.

50. Vasilev AD, Malashenko IS, Moiseev VF, Pechkovsky EP, Suljenko VK, Trefilov VI. (1978). Fracture mechanisms in particle-hardened Mo alloy with strong interface boundary. Problems of Strength. 1, 60-64, in Russian.

51. Vasilev AD, Lueft A, Perepoelkin AV, Firstov SA. (1979). Influence of temperature and rate of loading on structure of fracture surface of molybdenum, in: Wissenshaftliche Berichte, ZFW, <u>17</u>, II Soviet-German Symposium "Festkorperphysik und Werkstofforschung," 3-10, in Russian.

52. Vasilev AD, Pokhodnia IK, Trefilov VI, Firstov SA. (1981). Definition of effect-

ive surface energy at fractographical studies. Phys. Chem. of Mat. Treat. 3, 100-104, in Russian.

53. Vasilev AD, Gornaya ID, Moiseev VF, Pechkovskii EP, Ponomarev SS, Trefilov VI. (1982). Diagram of the strain-temperature and structural aspects of the fracture of molybdenum. Met. Phys. <u>4</u> 91-100, in Russian.

54. Vasilev AD, Moiseev VF, Firstov SA. (1984). Correlation between intergranular fracture and cleavage in bcc metals at brittle-ductile transition. Met. Phys. <u>6</u>, 68-72, in Russian.

55. Vergasov AN, Ribin VV, Solomko IV. (1976). Fracture peculiarities of molybdenum alloy, in: Physics of Brittle Fracture, part 1, IPM, Kiev, 72-76, in Russian.

56. Vergasov AN, Ribin VV. (1980). Structture peculiarities of microcrack initiation in molybdenum. Phys. Met. and Net. Sci. 46, 371-383, in Russian.

57. Wilsdorf HGF. (1979). In situ HVEM investigation of processes leading to fracture in metals. Krist. und Techn. <u>14</u>, 1265-1274.

#### Discussion with Reviewers

<u>Reviewer I</u>: Can you please define the Fridel-Orlov mechanism? Is it limited to bcc-materials? Why should striations of Fridel-Orlov mechanism originate? Author: The Fridel-Orlov mechanism is cleavage with periodic stops due to relaxation. It is limited to materials which cleave. Short description and scheme of this mechanism are given in /20/. According to the scheme which is quoted in Fig.16, plastic relaxation retards motion of cleavage crack. If velocity of crack is less some critical quantity, the crack stops suddenly. Tip of this crack is blunted. The crack will renew motion when tension stress will be increased. Continuous increasing of tensile stress which takes place at uniaxial tension results in periodic stops of crack. Periodic stopping and blunting of crack will produce striations on cleava-ge surface (fig.4c).



Fig.16. Plastic zones and their contours in successive stops of crack tip: a) surface of crack, b) cross-section. <u>Reviewer I</u>: Can you please define the <u>Cook-Gordon</u> mechanism? Is it limited to bcc materials?

bcc materials? <u>Author</u>: The Cook-Gordon mechanism is mechanism of blunting of crack tip by longitudinal stress ahead of crack /14/. It is valid for materials with interphase boundaries: particle-hardened materials, delaminated ones and composites. <u>Reviewer I</u>: Where, when and why do small dimples turn into large ones? <u>Author</u>: In particle-hardened materials, e.g., /5,45 and others/. At increasing of temperature, above  $T_t^{\mu}$ (fig.1b), dislocations do not apparently pile at particle-matrix interface due to transverse sliding. Particles are not delaminated from matrix. Alloy behaves as single-phase one. Origin of pores is not connected with particles because they are absent in dimples of fracture. Com-

are absent in dimples of fracture. Com-pare Figs. 13, 14. <u>Reviewer III</u>: In your paper you superimpose two different effects: a) The characteristic brittle-ductile transition which is shown by bcc and hexagonal metals and which depends on temperature, rate of loading, state of stress and the condition of the material. b) The tendency to intercrystalline fracture which mainly depends on the condition of the grain boundaries (especially precipitates, impurities and the like). Intercrystalline fracture may also occur at high temperature (liquid films on grain boundaries) and, in principle, all poly-crystalline metals and alloys could be concerned. Would you agree to distinguish clearly between these two effects? Author: Really, there are the intergra-nular fractures to be expected in polycrystalline materials. But the intergranular fracture, in materials which are apt to brittle-ductile transition, is temperature sensitive /54/. Manifestation of intergranular fracture depends on intergranular strength and its temperature dependence, and competition be-tween intergranular fracture mechanism and other ones.

Reviewer III: Can a plastic deformation by sliding or twinning precede the intercrystalline cracking? I don't think that such a mechanism occurs in general. <u>Author</u>: In principle, a plastic deformation is not necessary for fracture of materials with weak grain boundaries. But in materials with sufficiently high strength of grain boundaries, plastic deformation may be necessary to produce intergranular crack for following fracture. Scheme of this process was given by Zener (Zener C.(1948). The micro-mechanism of fracture, in: Fracturing of metals. ASM, Cleveland, <u>40</u>, 3-31). Fractographical evidence may be also given. Reviewer III: Did I understand you correctly that there should be no particles in the dimples after a tensile test at higher temperatures? That is quite unusual and contradicts the common experience. <u>Author</u>: If temperature dependences of flow stress and plastic characteristics of your particle-hardened alloys are similar Fig.1b, I think, it is possible to find some temperature range in limits of which there should be no particles in dimples of fracture. Reviewer I: Fig. 7g and 7h, what is a "usual" fracture of (which) Mo alloy? Author: "Usual" fracture is fracture after testing. I used this term to differentiate a fracture after testing from a fracture produced by longitudinal cleavage of fractured sample at low

temperature. <u>Reviewer I:</u> In the 'Ductile Fracture' section the author says, 'Wilsdorf et al. (text ref. 36, 57) have fractured samples in situ in the HVEM.'' In which manner? Thin foils?

Author: Wilsdorf et al. have investigated beryllium thin foil under uniaxial tension.

Reviewer I: V. Thien Reviewer III: G. Lange