REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From – To)*
30-03-2003	Final Report	20-Sep-01 – 28 Feb-03

4. TITLE AND SUBTITLE

Fabrication and Investigation of Large Billets of Ti-6Al-4V Alloy with Ultrafine-Grained Structure Using SPD-Processing.

5a. CONTRACT NUMBER
ISTC Registration No: 2125p

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

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5d. PROJECT NUMBER

5d. TASK NUMBER

5e. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
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8. PERFORMING ORGANIZATION REPORT NUMBER

N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

EOARD
PSC 802 BOX 14
FPO 09499-0014

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
ISTC 00-7048

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

This report results from a contract tasking Institute of Physics of Advanced Materials, Ufa State Aviation Technical University as follows: The present project aims to produce an ultrafine-grained (UFG) structure for the first time in large Ti-6Al-4V billets, using severe plastic deformation (SPD) processing through equal-channel angular (ECA) pressing. Special attention in this project will be devoted to the formation of a homogeneous UFG structure. The investigations will include modelling of plastic flow of the material during ECA processing, determination of factors influencing the localization of plastic strain and formation of an UFG structure, characterization of microstructure and phase composition of the alloy during ECA pressing, and the study of mechanical properties of the ECA-processed Ti-6-4 alloy.

15. SUBJECT TERMS
EOARD, Materials, Metallurgy & Metallography

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18, NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
UNCLAS	UNCLAS	UNCLAS	UL		Charles H. Ward, Lt Col, USAF
					19b. TELEPHONE NUMBER *(Include area code)* +44 (0)20 7514 3154

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39-18

ISTC 2125p

Final
Project Technical Report
of ISTC 2125p

Fabrication and investigations of large-sized billets out of Ti-6Al-4V alloy with ultrafine-grained structure using SPD-processing

I. Project Activities Report

(From 1 September 2001 to 28 February 2003 for 18 months)

Ruslan Zufarovich Valiev
(Project Manager)
Ufa State Aviation Technical University

March 2003

Vice-rector USATU, Professor **V.S. Zhernakov**

Project manager, Professor **R.Z.Valiev**

This work was supported financially by European Office of Aerospace Research and Development and performed under the contract to the International Science and Technology Center (ISTC), Moscow and EOARD.

Fabrication and investigations of large-sized billets out of Ti-6Al-4V alloy with ultrafine-grained structure using SPD-processing
(From 1 September 2001 to 28 February 2003 for 18 months)

Ruslan Zufarovich Valiev
(Project Manager)
Ufa State Aviation Technical University*

The objective of this project is to develop equal channel angular pressing (ECAP), as method of severe plastic deformation (SPD), for fabrication and investigation of large-sized billets out of VT 6 (Ti-6Al-4V) alloy, containing ultrafine-grained (UFG) structure with grain size less than 0.5 μm).

Project summary: Within the frames of the project complex investigations, aimed at processing of large-sized billets out of VT 6 (Ti-6Al-4V) alloy with UFG structure, have been performed.

Formation of homogeneous UFG structure with grain size less than 0.4 μm in billets ∅ 20 mm by ECAP technique and subsequent warm upsetting has been shown. It has been demonstrated that such treatment results in considerable increase of strength properties of the alloy. Their values reach ultimate tensile strength of 1450 MPa and yield stress of 1420 MPa.

Using computer and experimental simulation of ECAP process, the possibility of its scaling for fabrication of large-sized billets of ∅ 40 and 120 mm and the subsequent upsetting has been developed.

Keywords: titanium alloy Ti-6Al-4V (VT6), ultrafine-grained structure, large-sized billets, severe plastic deformation, equal channel angular pressing.

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2. Project Goals

Development of equal channel angular pressing (ECAP), as the method of severe plastic deformation (SPD) for fabrication and investigation of large-sized billets out of VT6 (Ti-6Al-4V) alloy with ultrafine-grained (UFG) structure (with grain size less than 0.5 µm).

3. Scope of Activities and Research- Technical Approach

VT6 alloy, the Russian analogue of industrial Ti-6Al-4V alloy, was used as material for investigations. Development of the experience of Ufa researchers in the sphere of ECAP treatment of hard-to-deform materials, in particular, in Ti materials, served as methodological basis for the investigations.

As a result of computer and experimental simulation we established unit loads and temperature intervals of pressing, as well as the number of other important ECAP parameters, such as deformation rate, number of passes and pressing routes. Optimization of the defined regimes was realized by structural investigations and tests of mechanical properties of processed billets. Processing of billet samples, characterization of their structure and mechanical properties were performed by scientists and researchers of IPAM, USATU. The equipment and specialists of technical center NKTB ”Iskra” were also engaged for fabrication of large-sized billets ∅120 mm.

4. Resume of Project Technical Report

The present final report represents the results of investigations that demonstrate for the first time the possibility of using equal channel angular pressing (ECAP) to process large-sized Ti-6Al-4V billets with ultrafine grain size (UFG) less than 0.5 µm.

The problem of processing bulk billets ∅ 20 mm and more with UFG structure out of Ti-6Al-4V alloy by ECAP still remains highly topical and that is connected with great complexity of carrying out severe deformation of the given alloy. This task requires integrated approach and overcoming of many problems related to materials science and forming for hard to deform materials, that have been investigated within the frames of the present project:

a) a) Numerical simulation and experimental studies of ECAP of the alloy.
b) Designing and fabrication of the ECAP die-sets for processing billets ∅ 20 mm, ∅ 40 and ∅120 mm. Optimization of ECAP regimes using the results of the experiments.
c) ECAP processing the given billets with UFG structure and then their upsetting to fabricate the billets in the form of disks.
d) г Investigation of microstructure homogeneity, phase composition and mechanical properties in the processed large-sized billets.

The results on designing and fabricating of ECAP die-sets, ECAP modeling, optimization of ECAP regimes as well as the investigation results of microstructure and mechanical properties of processed samples ∅ 20 mm out of VT-6 alloy, the Russian analogue of Ti-6Al-4V alloy (ASTMF 136), are presented in the given report.

Regimes of multipass ECAP within the temperature range 600-800°C were defined for processing UFG structure and enhancing of mechanical properties in large-sized billets out of VT6 alloy.

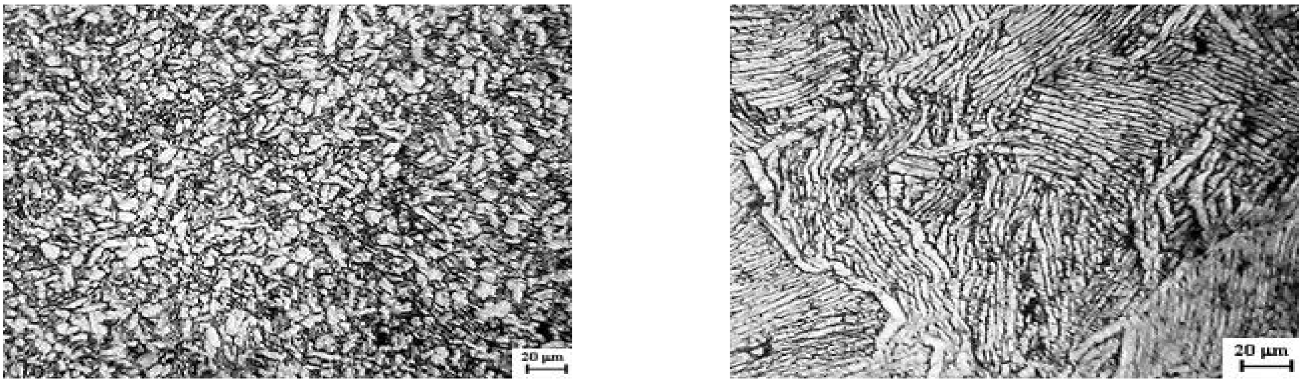
The final project goal was processing large-sized billet ∅200x100 mm with UFG structure, that supposed ECAP of billets ∅120x450 mm, subsequent machining up to ∅120x220 mm and then its warm upsetting up to ∅200x100 mm.

In the work the VT-6 alloy was used that is a Russian analogue of the commercial Ti-6Al-4V alloy (ASTM F 136). The alloy was manufactured and supplied by Verkhne-Saldinsky metallurgical production association (AO VSMPO) in the form of hot-rolled bars ∅ 25 mm, ∅ 40 mm and 150 mm. The temperature of polymorphous $\alpha \rightarrow \beta$ transformation is 990°C. The chemical composition of the alloy is given in Table 1.

Table 1. Chemical composition of VT6 in weight % (actual and in accordance with the standard)

	Ti	**V**	**Al**	**C**	**N**	**Fe**	**Si**	**H**	**O**
GOST	Base	3.5…5.3	5.3…6.8	0.10	0.05	0.60	0.10	0.015	0.20
Act.	Base	4.8	6.6	0.02	0.01	0.13	0.04	0.002	

The initial microstructure was globular for rods ∅25 mm (Fig.1a) and lamellar for rods ∅40 and 150 mm (Fig.1b).



a) b)

Fig. 1. Optical microphotography. The structure of the initial alloy: a) globular; b) lamellar

The performed investigations included simulation of ECAP process, the experiments on workability of the billets in various structural states during ECAP, and also structural characterization, microhardness measurements and tensile tests, aimed at definition of regimes, forming a homogeneous UFG structure in the samples out of VT6 alloy.

ECAP was carried out with billets ∅ 20 x 70 mm in three structural states:

1) as received state;
2) quenched from (α+β) area at 950°C;
3) quenched from β area at 1010°C.

The study of structure uniformity in the volume of the deformed samples, and also morphology and size of phases, forming during ECAP process for various conditions, have shown that the initial state of rods ∅25 mm is characterized by the fine-grained globular structure mainly (Fig. 1,a). The structure peculiarity is the presence of rather large globules up to 6 μm of the primary β - phase and plate-like dispersed precipitation of the secondary α - phase.

Multipass ECAP with 13 passes at billet temperature 700°C causes a notable refinement of the initial structure in the cross section up to grain size about 0.5 μm. The slight structure elongation in the direction of the rolling typical for the initial alloy practically disappears. Metallographic analysis, performed in three arbitrary cross sections along the rod's length, has shown high structure homogeneity of deformed samples in longitudinal and cross sections.

As it is seen, after ECAP at the billet temperature of 700 °C the SAED pattern taken off from the areas of 2 μm^2 looks like rings with the numerous spots, randomly distributed on them. Such electron-diffraction pattern can testify to ultrafine-grained structure formation where high-angle boundaries are present. The bright-field image testifies to the grain size less 0.5 μm. The microstructure is characterized by high density of various defects (dislocations, grain/subgrain boundaries) and high internal stresses.

The results of tensile tests of VT6 alloy in various states, are presented in Table 2. The results testify that ECAP for all investigated states leads to increasing of strength characteristics of UTS and YS by 20 % in comparison with the initial state of the alloy. At this the elongation before failure though decreases, but it remains high enough.

Table 2. Mechanical properties of VT6 alloy in various states

#	State	UTS, MPa	YS, MPa	δ, %
1	Initial (as received)	970	900	20
2	1 + ECAP at 700 °C, 13 passes	1160	1110	12
3	2 + upsetting at 650 °C – 1 hour	1140	1100	8
4	2 + upsetting at 600 °C, ε = 55%)	1450*	1420*	11*

for states 1-3 the samples ∅ 5 x 25 mm were tested;
* for state 4 the samples 5 x 7 x 2.5 mm were tested.

The results of microstructure investigations after ECAP and additional warm upsetting have shown that a mean grain size even decreases slightly as compared to ECAP state and amounts to 0,4 µm. The grain form is equiaxed mainly both in longitudinal and cross sections with regard to the upsetting direction The image of electron-diffraction patterns with many spots along circles testifies that the forming structure is the structure of grain type with high-angle misorientations The observed spreading of spots in electron diffraction patterns testifies the presence of internal stresses. Lattice dislocations are obviously considered to be the source of these stresses.

The results of mechanical tests of ECAP alloy after upsetting (Table 2.) show that UTS of ECAP alloy after upsetting is higher by 27 % than the initial ECAP. It seems connected with the formation of more equiaxed structure with smaller grain size. The achieved UTS (1450 MPa) approaches to record level for such type of titanium alloys. To process ECAP large-sized billets ∅120 mm it was necessary to solve a number of technical and scientific problems, connected both with forming of billets and a formation of homogeneous UFG structure.

Technical aspects of the problem included designing of safe die-set and adjustment of regimes, consisting in optimization of pressing temperature and tribological properties of the process as well as in searching of pressing route. To reduce expenses a well-known approach, used in metal forming research, was applied. This approach consists in keeping to geometric similarity on 1: 3 scale. At that we chose billet diameter of 40 mm for investigations of scaling problems. The main goal of these investigations was processing of integral UFG samples without failure out of the alloy after considerable (e>5) strain. Scientific aspects included also the investigation of the influence of initial structural state of the billets before ECAP on their workability and formation of UFG structure.

Deformation of billet ∅40 mm, using multipass treatment at die-set temperature up to 500 °C and billet temperature up to 850 °C was realized due to fabrication of die-set out of heat-resistant steels and special construction.

ECAP of billets ∅40 mm, using regimes for billets ∅20 mm, has revealed low processing deformability and accumulated strain, as the billets at 700 °C failed already after 4-6 passes. In this case ECAP does not lead to considerable structure refinement. Microstructure is mainly platelet, plate size is about 500 nm in width. Grain structure is not formed.

Thus, despite of the possibility of processing ECAP billets ∅40 mm at 700 °C, it was impossible to secure accumulated strain e > 6 without billet failure and to process UFG structure. To our opinion, it is connected with lamellar structure in the initial billet. Therefore, we investigated the possibilities of enhancing workability of large-sized billets, connected with transformation of lamellar structure into globular one before and during ECAP process.

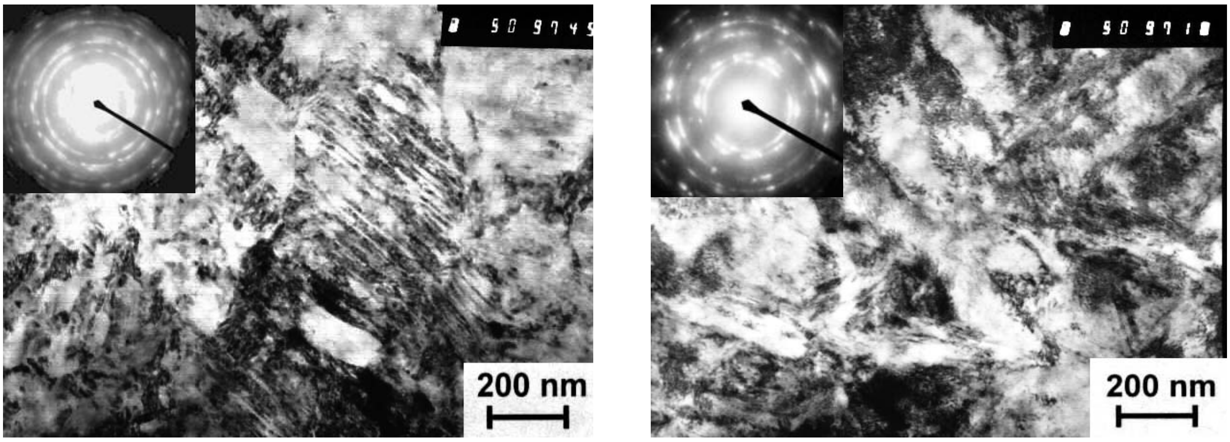
Preliminary thermal treatment - annealing at 950°C for 6 hours with the subsequent cooling in furnace enabled to process the mixture of globular and lamellar structures with grain size of α-phase less than 10 μm. Nevertheless, the subsequent ECAP of billet with such mixed structure resulted in its failure after the first pressing route. However, some structure refinement was also observed.

ECAP at elevated temperatures

ECAP was performed at step-like decreasing of temperature by 50°C after every two pressing passes, starting with 850 down to 700°C. It allowed to attain accumulated strain e = 5.6 without billet failure. However, in accordance with the data of transmission electron microscopy, such treatment did not result in formation of UFG structure, despite the fact that the structure was noticeably refined. Bending of α-phase plates occurred as well during deformation process. Some part of those plates was subjected to spheroidization.

The most effective treatment was 4 ECAP passes at 850°C +annealing at 800°C for 2 hours + 8 ECAP passes at 700°C.

Structure analysis of these samples has shown that such treatment does lead to noticeable microstructure refinement in the presence of many areas with UFG structure (Fig.2). Numerous sports at SAED pattern rings testify predominance of high-angle boundaries. Some spreading of sports is also typical of this state.



a) b)

Fig. 2. Structure of VT-6 alloy ∅40 mm after 4 ECAP passes at 850°C + annealing 800°C, 2 hours + 8 passes at 700°C in cross section (a) and in longitudinal section (b).

Tensile tests of the samples with basis of 5 mm and section of 2.5x0.65 mm^3, cut out in cross direction of ECAP billet, have shown that mechanical properties of the alloy in the given state amounted to: ultimate tensile strength - 1190 MPa, yield stress – 1130 MPa, elongation – 12 %. These values practically coincide with the values of these properties for ECAP ∅20 mm with globular initial structure (Table 2, State 2).

As a result of performed investigations we processed integral billets ∅40x150 mm (Fig. 3) with UFG structure and level of properties, similar to which attained on samples ∅20 mm. This fact testifies the possibility of successful scaling of ECAP VT6 alloy.

Fig. 3. The image of ECAP sample ∅40 mm without damage, processed by ECAP at 850°C (4 passes) +annealing 800°C -2 hours + ECAP at 700°C (8 passes).

For processing of large-sized billets ∅ 200 x 100 mm, including ECAP and the subsequent upsetting computer simulation by finite elements method (FEM) was performed. The obtained data enabled to choose the necessary length of the initial billet for ECAP, which is equal to 450 mm, to calculate pressing load that amounted to 370 tons. The last parameter predetermined the choice of pressing equipment.

Initial billets ∅120 mm, as compared to billets ∅40 mm, had more coarse lamellar structure, in which the size of former β grains was about 150-300 µm.

Therefore, multifold forging of the billets with changing of deformaton axis at 850°C and subsequent annealing at 800°C were used to refine and form globular structure and to enhance, correspondingly, workability of the alloy. The conducted treatment, performed by hydraulic press with loading 1600 tons (Fig. 4) allowed to refine noticeably the structure in large-sized billet and to enhance potentially its workability.

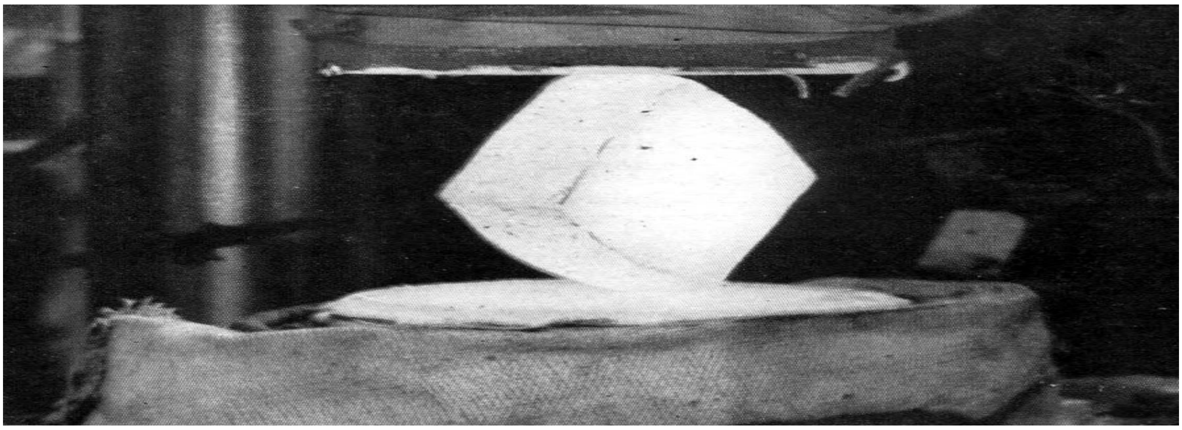


Fig. 4. Process of multifold forging of the billet out of VT-6 alloy before ECAP.

Analysis of stress-strain state of billet, based on data of computer simulation, enabled to perform calculations for designing die-set for ECAP of billets ∅ 120 mm. It revealed that it is necessary to ensure elevated strength margin due to formation of double banding of the forming matrix.

However, the weight of such die-set amounted to 5 tons, and its fabrication costs exceed the project financing. Therefore, a die-set with only single banding was fabricated. Using the given die-set, ECAP of billets ∅120 mm was performed. The structure of these billets was prepared following the technique, described in part 4.2. As a result in two billets one ECAP pass was performed. During the second pass of pressing in the first billet the pressing stress increased abruptly due to local adhesive gripping (welding) of the billet and die-set. It is probably connected with the scaling effect, i.e. with higher contact temperature on the boundary "billet-insertion" due to greater mass of the billet as compared to billet ∅40 mm. Several cracks occurred in the most loaded central part of insertion due to enhancing of friction and pressure forces.

To process the final form of the forged piece ∅200x100 mm we conducted singe-axis warm upsetting of ECAP billet with relative strain 60%. The initial billet before upsetting was of ∅118 x 276 mm. The upsetting of the billet was performed at 700°C on flat blocks out of heat-resistant steel in isothermal conditions on the hydraulic press by loading of 1600 tons (Fig. 11). The maximum upsetting loading amounted to 1230 tons. The averaged accumulated strain amounted to $e = 0.85$. The final form of the billet ∅200×100 mm.

Metallographic analysis of the processed billet has shown that even after one ECAP pass and subequent warm upsetting the microstructure became more dispersed and homogeneous, but for fabrication of UFG structure it is important to perform multipass ECAP.

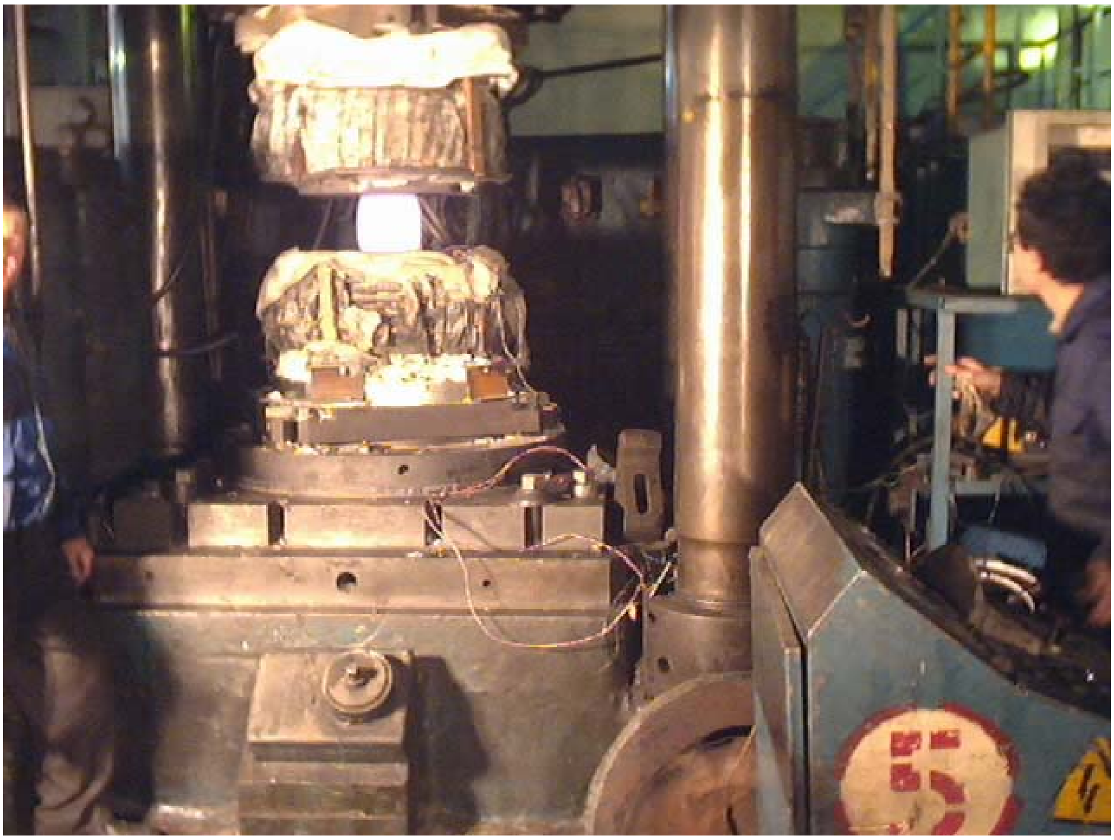


Fig.5. Upsetting of billet out of VT 6 alloy ∅120 nm on press by loading of 1600 tons.

The technical task, draft project (Fig. 23) and working drawings of the ECAP die-set ∅ 20 mm with backpressure for hard-deforming alloys are worked out in accordance with the statement of work (SOW).

The ECAP die-set was fabricated, tested and dispatched to the partner.

The total weight of the die-set amounts to 200 kg. The dispatch was effected through ISTC.

At present the die-set has been tested by the customer and positive conclusion has been made.

5. Project Results

5.1. List of published articles without their resumes

1. V. V. Stolyarov, R. Z. Valiev, Structure-phase Transformations and Properties in Nanostructured Metastable Alloys processed by Severe Plastic Deformation, 2nd International Conference on nanomaterials by severe plastic deformation, Austria, Vienna December 9-13, 2002, will be published in a hardcover proceedings by Wiley-VCH.
2. R.Z. Valiev, Paradoxes of Severe Plastic Deformation, Proceedings of the 2nd International Conference on Nanomaterials by Severe Plastic Deformation: Fundamentals – Processing – Applications (9-13 December 2002, Vienna, Austria), to be published in Advanced Materials Engineering.

5.2. List of conference reports without their resumes

1. V. V. Stolyarov, R. Z. Valiev, Bulk nanostructured alloys from severe plastic deformation, International Workshop "New Production Technologies and Materials", Portugal, Lisbon, December 2-5, 2002.

2. V. V. Stolyarov, R. Z. Valiev, Structure-phase Transformations and Properties in Nanostructured Metastable Alloys processed by Severe Plastic Deformation, 2nd International Conference on nanomaterials by severe plastic deformation, Austria, Vienna December 9-13, 2002.

3. R.Z. Valiev, Paradoxes of Severe Plastic Deformation, The 2nd International Conference on Nanomaterials by Severe Plastic Doformation: Fundamentals – Processing – Applications (9-13 December 2002, Vienna, Austria), invited.
4. R.Z. Valiev, Bulk Nanostructured SPD Materials with Unique Properties, The 2003 TMS Annual Meeting (2-6 March 2003, San Diego, USA), invited.

5.3. Patent and Author's Rights Information

Russian patent № 2181314. Apparatus for treatment of metals by high deformation and pressure / Raab G.I., Kulyasov G.V., Polozovsky V.A., Valiev R.Z.- Published on 20.04.2002.

The know-how is aimed at improvement of device construction for ECAP of materials. The features of constructive fulfillment of pressing channels, aimed at enhancing deformation homogeneity and decreasing pressing loads, are reflected in it.

6. Foreign Collaborators' s Partering

1. Prof. S.L. Semiatin, Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/ MLLM, Wright- Patterson Air Force Base, OH 45433-7817, USA.

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2. Prof. H.J. Rack, Sc. D., FASM. Department of ceramic & materials engineering.
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6.1. Research materials change

During Project implementation intensive change of pilot samples, plans and reports on investigation results was effected.
The samples out of VT6 alloy, subjected to different ECAP regimes for investigation of the structure, mechanical and tribologic properties, were dispatched to the partner.
The die-set with backpressure for ECAP of hard-to-deform alloys was designed, fabricated and dispatched to Prof. Semiatin.

6.2. Signing of protocols

none

6.3. Partnering Research Investigations

Partnering investigations of tribologic properties of UFG VT6 alloy, processed by ECAP ∅20 mm, are currently carried out with Prof. H.J. Rack.

6.4. Business trips to foreign collaborators' organizations/ business trips of collaborators' representatives

none

6.5. Seminars, thematic conferences, organized by Project participants

Participation of E.I. Andrianova (translator) in seminar "Effective R&D project management", organized by the Urals Scientific- Educational Center of Innovation Business, ISTC, Ekaterinburg, January, 17-18, 2003.

6.6. Partnering Participation in International Conferences

From 13.02 till 22.02.2002 R.Z. Valiev, V.V. Stolyarov, G.I. Raab represented their reports at TMS-conference, Seattle.

7. Partnering with NIS subcontractors

The equipment and specialists of technical center NKTB ”Iskra” (Ufa) were also engaged for fabrication and upsetting of large-sized billets ∅120 mm.
The meeting with specialists of South- Ural State University (Chelyabinsk), experts in the field of production treatment of large-sized billets out of titanium alloys, was conducted.
Fabrication of ECAP die-set was carried out in cooperation with NVP “Link”, Ufa.

8. Technology Implementation Plan

Application of the Project results in next work

The project result is development of ECA pressing for processing UFG structure with grain size less than 0.5 μm in VT6 (Ti6-Al-4V) alloy. The performed investigations revealed problems of ECAP scaling during processing of large-sizeв billets with UFG structure. These problems are connected with low workability of the alloy with lamellar structure. Treatment regimes, which enable to transform lamellar structure into globular one and as a result to successfully realize multipass ECA pressing, were defined. It makes possible to process large-sized billets out of the alloy with UFG structure and improved properties.

Prospects of research investigations/ developed technologies in future development

Formation of UFG structure results in considerable increase of mechanical properties of VT6 alloy. The given alloy is widely used in aerospace industry for manufacture of turbine disks for gas turbine engines. Performed investigations formed scientific fundamentals for fabrication of large-sized billets with UFG structure, which are suitable for wide constructive application in propulsion engineering.

Commercial potential of the Project results

Application of alloy with UFG structure and enhanced mechanical properties is highly perspective for increase of operating characteristics of gas turbine engines, such as working time, take-off weight and others.

Patents and Author's Rights

As a result of project activities some original results have been received. They are connected with regimes of thermo-mechanical treatment for transformation of the initial lamellar structure into globular one, as well as with ECAP regimes. The obtained results will be used for submission of partnering application for patent.

Russian patent № 2181314. Apparatus for treatment of metals by high deformation and pressure / Raab G.I., Kulyasov G.V., Polozovsky V.A., Valiev R.Z.- Published on 20.04.2002.

The know-how is aimed at improvement of device construction for ECAP of materials. The features of constructive fulfillment of pressing channels, aimed at enhancing deformation homogeneity and decreasing pressing loads, are reflected in it.

Appendix: "Technology Implementation Plan"

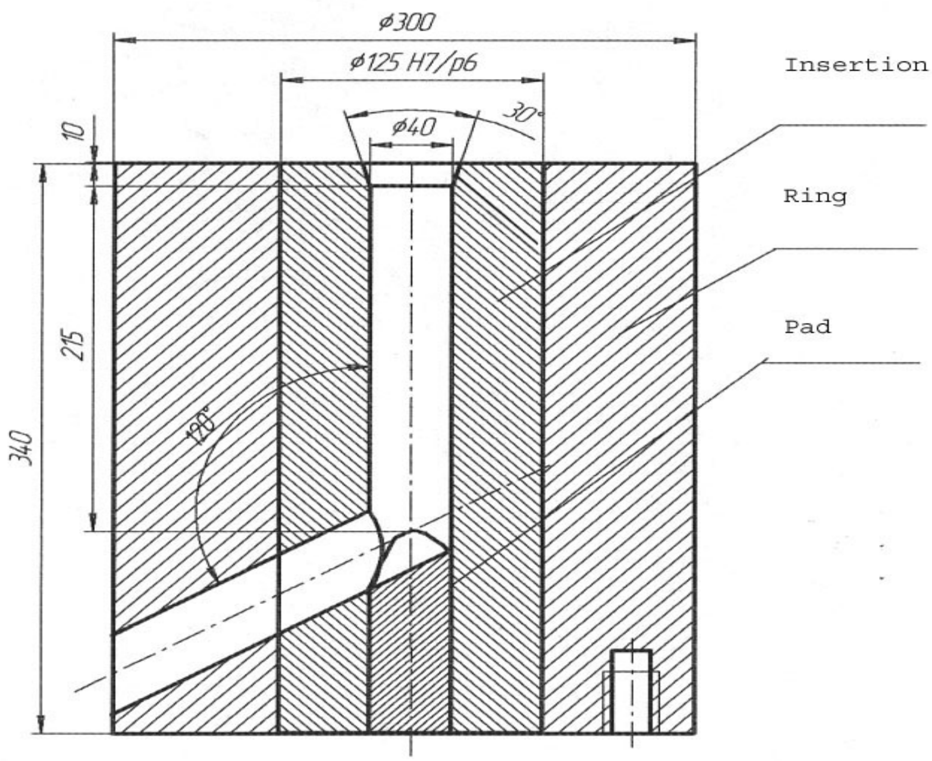
3. Processing and investigation of ECAP billets Ø40 mm

To process ECAP large-sized billets ∅120 mm it was necessary to solve a number of technical and scientific problems, connected both with forming of billets and a formation of homogeneous UFG structure. Automatic transmission of the obtained results of experiments of ECAP billets ∅ 20 mm to the full-scale ECAP process of billets ∅ 120 mm was impossible due to structure difference in the initial billets and as a result to differences in processing regimes. Designing of special die-set for ECAP ∅ 120 x 450 mm is also a problem. This die is much heavier than the die-set ∅ 20 mm - 5 tons and 70 kg, correspondingly.

Technical aspects of the problem included designing of safe die-set and adjustment of regimes, consisting in optimization of pressing temperature and tribological properties of the process as well as in searching of pressing route. Performance of such investigation on billets ∅ 120 mm is very labor-consuming and it exceeds the project financing. Therefore, a well-known approach, used in metal forming research, was applied. This approach consists in keeping to geometric similarity on 1: 3 scale [16]. At that we chose billet diameter of 40 mm for investigations of scaling problems. The main goal of these investigations was processing of integral UFG samples without failure out of the alloy after considerable (e>5) strain. The die-set construction was worked through as well. Its principles were used for fabrication of ECAP die-set ∅ 120 mm. Scientific aspects included also the investigation of the influence of initial structural state of the billets before ECAP on their workability and formation of UFG structure.

Deformation of billet ∅40 mm, using multipass treatment at die-set temperature up to 500 °C and billet temperature up to 850 °C was realized due to fabrication of die-set out of heat-resistant steels and special construction that consists of several parts (Fig. 7), see also [17]. The best tribologic pressing conditions were obtained when lubrication “Angelina” with lamellar graphite stuff was used. Channel intersection angle amounted to 120°. Such die-set enabled to successfully realize ECAP of billets out of the alloy with initial lamellar structure, where initial pressing was carried out at relatively high temperature 850°C (see below).

a)



b)

Fig .7. Scheme of the die-set with channel intersection angle 120°C for ECAP billets out of VT-6 ∅40 mm.

3.1. Investigation of workability of the alloy with various initial structure

As it was noted in part 2, the initial alloy rods had sufficiently different structural states. There were globular microstructures in rods ∅20 mm and lamellar ones in rods ∅40 mm and 150 mm. At first the role of the initial microstructure of the alloy was investigated by upsetting of small samples on flat panes. Cylindrical samples ∅8x10 mm were cut out of billets with globular and lamellar structures. Then the samples were upset at 600 and 700 °C. The upsetting was performed on universal testing machine 1231У-10 with crosshead speed of 1mm/min.

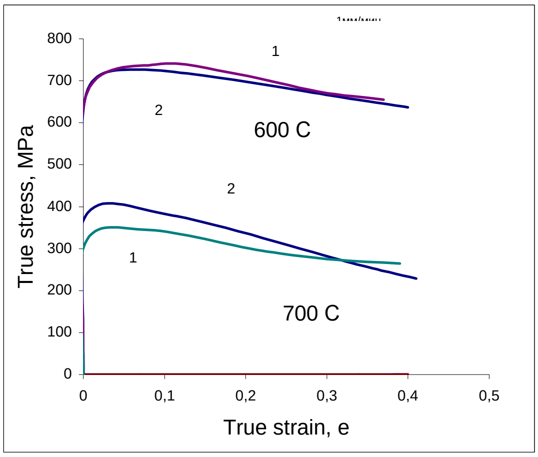
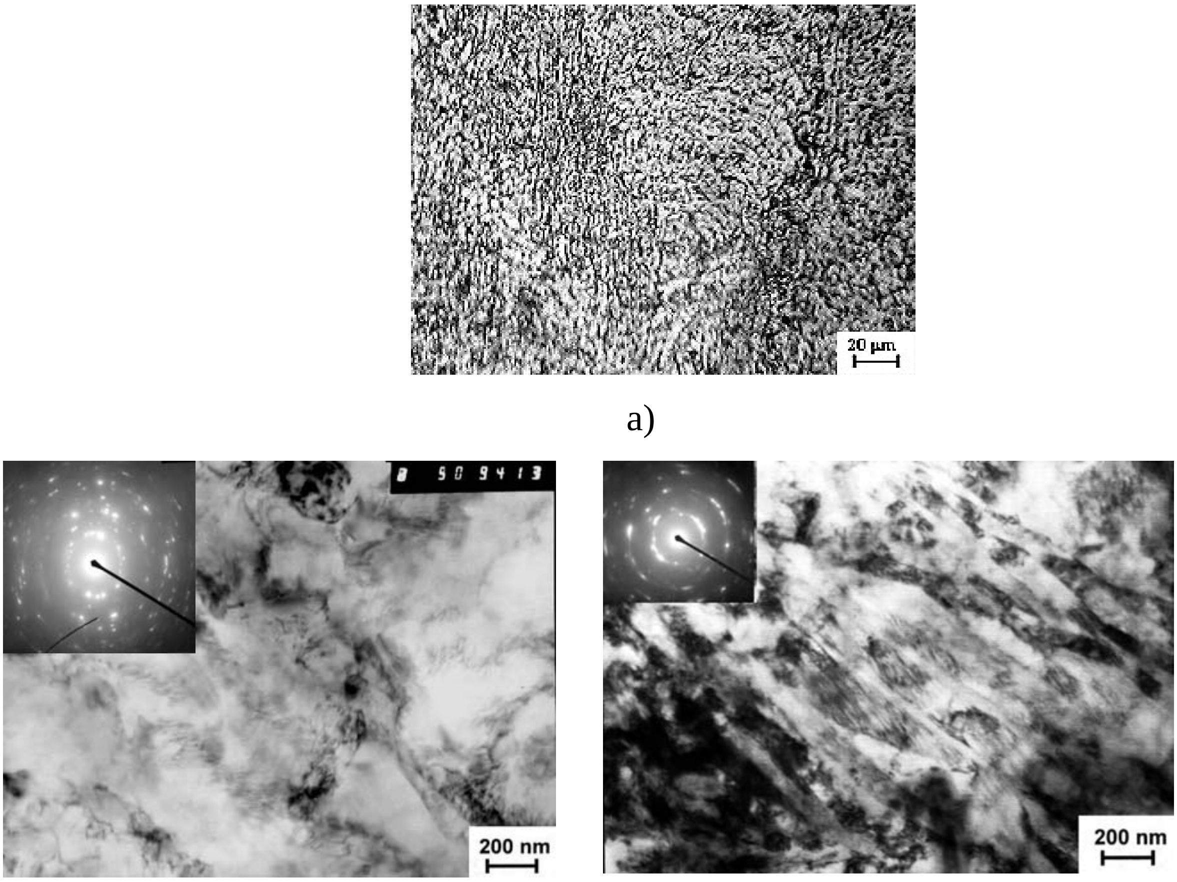


Fig. 8. Stress-strain curves during upsetting of the alloy at 600 and 700 °C in the initial state with globular (1) and lamellar (2) structure

As it can be seen in Fig.8, flow stresses in both states of the alloy reach the maximum at e ≈ 0.05. Then they rapidly decrease at enhancing of strain degree. Peak stresses and softening effect (difference of the maximum and minimum flow stress) are the function of deformation temperature and structure. At 600 °C flow stresses and softening effect (85 MPa) slightly depend on structure type. At the same time the peak stress and softening effect (170 MPa) at 700 °C for lamellar structure are higher than for globular one. When deformation temperature elevates from 600 up to 700 °C, flow stresses for both structure types abruptly decrease nearly twice.

These investigations of workability of the initial alloy during upsetting did not reveal obvious advantage (for example, elevated cracking resistance) of globular structure at 700 °C over lamellar one. However, the other fact was established during ECAP of billets ∅40 mm.

In this case low processing deformability and accumulated strain were revealed during ECAP of these billets, using regimes for billets ∅20 mm (see part 2), the billets at 700 °C failed already after 4-6 passes. As it can be seen in Fig. 9. ECAP does not lead to considerable structure refinement. Microstructure is mainly platelet, plate size is about 500 nm in width. Grain structure is not formed.



a)

b) c)

Fig. 9. Microstructure of the alloy after ECAP ∅40 mm at 700 °C, 6 passes: optical metallography (a); transmission electron microscopy in cross section (b) and longitudinal section (c).

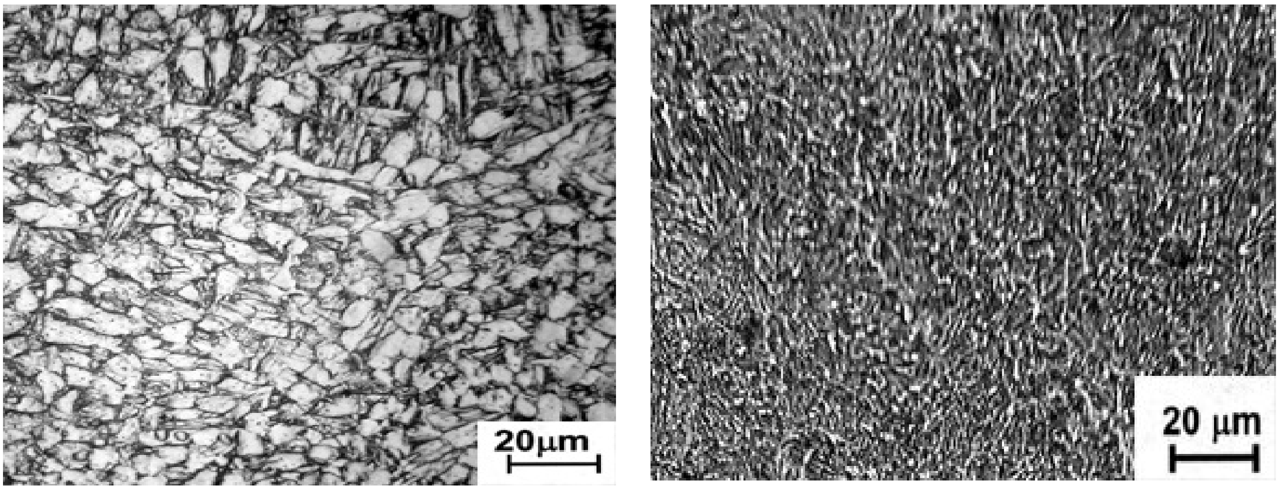
Thus, despite of the possibility of processing ECAP billets ∅40 mm at 700 °C, it was impossible to secure accumulated strain e > 6 without billet failure and to process UFG structure. To our opinion, it is connected with lamellar structure in the initial billet. Therefore, we investigated the possibilities of enhancing workability of large-sized billets, connected with transformation of lamellar structure into globular one.

3.2. Optimization of ECAP billets ∅ 40 mm

We investigated two methods of transformation of lamellar microstructure into globular one by preliminary treatment of the initial structure in the alloy before ECAP.

Preliminary thermal treatment

To realize this task the initial billets out of rods ∅40 mm were subjected to thermal treatment – annealing at 950°C for 6 hours with the subsequent cooling in furnace. This treatment enabled to process the mixture of globular and lamellar structures with grain size of α-phase less than 10 μm (Fig.10 a). Nevertheless, the subsequent ECAP of billet with such mixed structure resulted in its failure after the first pressing route. However, some structure refinement was also observed (Fig. 10 b.)



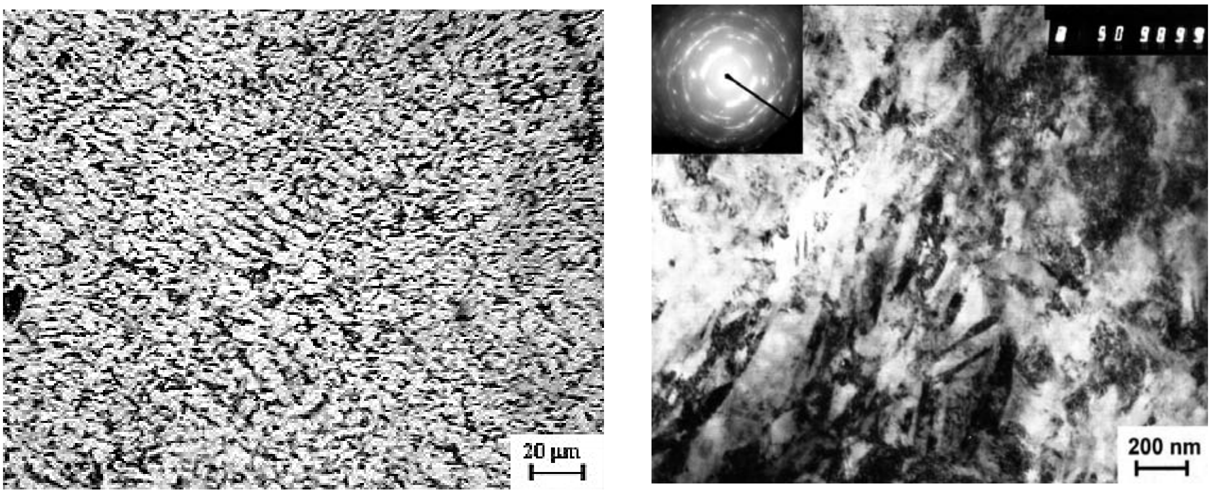
a) b)

Fig. 10. Structure of VT-6 alloy ∅40 mm after annealing at 950°C, 6 hours (a) and subsequent ECAP (b) at 700°C, 1 pass

ECAP at elevated temperatures

Other experiments were aimed at searching of the regimes, that secure the formation of globular structure and its refinement during ECAP process. The idea of this treatment is based on attempt to imitate well-known thermomechanical treatment. This regime is used in standard processing titanium semi-finished articles at hot deformation within α+β area.

In this connection there were performed ECAP at *step-like decreasing of temperature* by 50°C after every two pressing passes, starting with 850 down to 700°C. It allowed to attain accumulated strain e = 5.6 without billet failure. However, in accordance with the data of transmission electron microscopy, such treatment did not result in formation of UFG structure, despite the fact that the structure was noticeably refined. (Fig. 11a,b). Bending of α-phase plates occurred as well during deformation process. Some part of those plates was subjected to spheroidization.

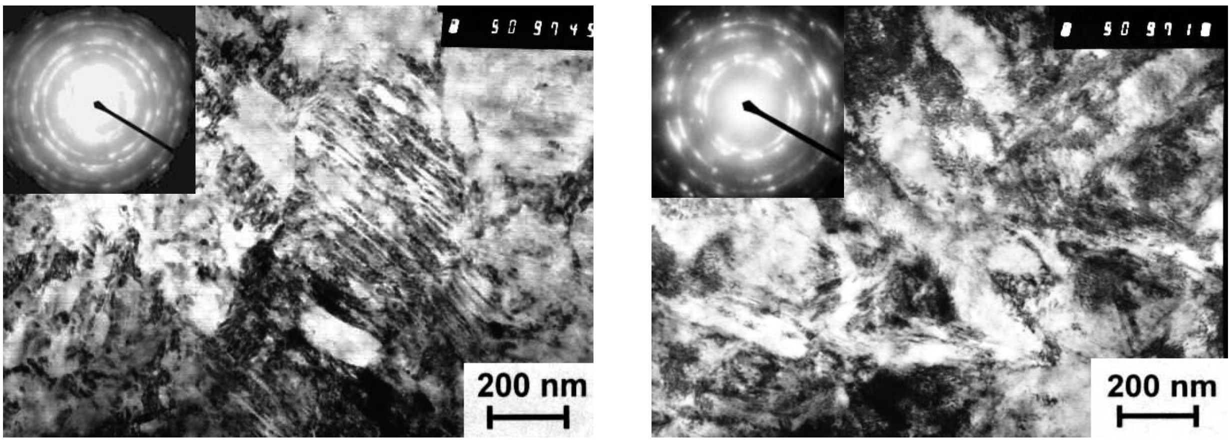


a) b)

Fig. 11. Structure of VT-6 alloy after ECAP 40 mm within the temperature range of 850-700°C. Optical metallography (a), TEM (b)

Therefore, we used one more treatment, namely, 4 ECAP passes at 850℃ +annealing at 800℃ for 2 hours + 8 ECAP passes at 700℃.

Structure analysis of these samples has shown that such treatment does lead to noticeable microstructure refinement in the presence of many areas with UFG structure (Fig.12). Numerous sports at SAED pattern rings testify predominance of high-angle boundaries. Some spreading of sports is also typical of this state. In a few cases mixed structure, consisting of plates and fragments, was observed as well. Substructure formation was revealed within the plates.



a b

Fig.12. Structure of VT-6 alloy ∅40 mm after 4 ECAP passes at 850°C + annealing 800°C, 2 hours + 8 passes at 700°C in cross section (a) and in longitudinal section (b).

Tensile tests of the samples with basis of 5 mm and section of 2.5x0.65 mm^3, cut out in cross direction of ECAP billet, have shown that mechanical properties of the alloy in the given state amounted to: ultimate tensile strength - 1190 MPa, yield stress – 1130 MPa, elongation – 12 %. These values practically coincide with the values of these properties for ECAP ∅20 mm with globular initial structure (Table 3, State 2).

As a result of performed investigations we processed integral billets ∅40x150 mm (Fig. 13) with UFG structure and level of properties, similar to which attained on samples ∅20 mm. This fact testifies the possibility of successful scaling of ECAP VT6 alloy.

Fig. 13. The image of ECAP sample ∅40 mm without damage, processed by ECAP at 850°C (4 passes) +annealing 800°C -2 hours + ECAP at 700°C (8 passes).

4. Development of ECAP of billets ∅120 mm

The results of ECAP process of billets ∅ 20 and 40 mm were used for development of processing large-sized billets out of VT-6 alloy with UFG structure. However, large sizes of the billets and their low workability, due to lamellar character of the initial structure of the billets, predetermined the application of special processing regimes. Computer simulation was used to increase workability and to analyze ECAP process of billets ∅120 mm [18,19 ,20]. The obtained data allowed to choose special pressing and thermal equipment, as well as to define the most probable regimes of processing large-sized UFG billets. These works are described below.

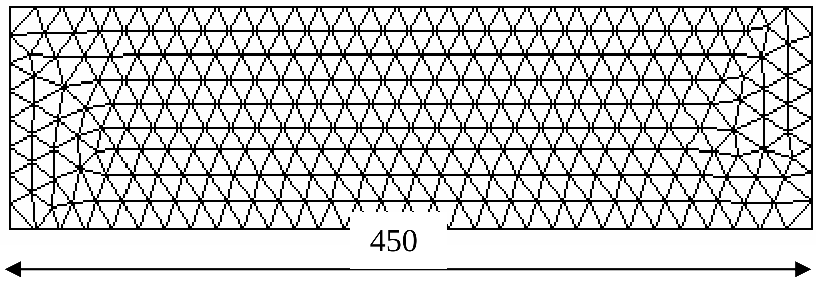
4.1. Computer simulation of ECAP billets ∅ 120 mm

The main goal of computer simulation of ECAP in the billets ∅ 120 mm, heated up to 700 and 800°C, was the definition of the following parameters: strain stress of the billet, pressing stresses and normal contact stresses of the most loaded elements of the experimental die-set, matrix and puncher. These parameters are determinant for designing of experimental die-set for ECAP, the choice of pressing equipment and processing of non-damage billets. The billet model ∅ 120 mm out of VT-6 alloy after one ECAP pass at 700 ^{0}C is given in Fig. 14. The analysis shows that in end parts of the billet (areas 1 and 2) the level of true accumulated strain does not exceed e= 0.5. At that the front end of the billet has more stretched non-homogeneous section. The central part of the billet (area 3), constituting about 2/3 of billet length, has accumulated strain e= 0.77 It is agreed to the value e=0.7, calculated on the formula of true strain during ECAP [21]. However, low contact layers of the billet (areas 4 and 5) have higher values of true accumulated strain e=1.05 and e=1.33. It differs to some degree from the data obtained at computer simulation of ECAP process of billets ∅ 20 mm out of VT-6 alloy when the true accumulated strain in low contact area amounts to e= 0.89 (Fig. 2). The data obtained by net method for the billets ∅ 20 mm after ECAP [22] also testify the decrease of accumulated strain in low contact area.

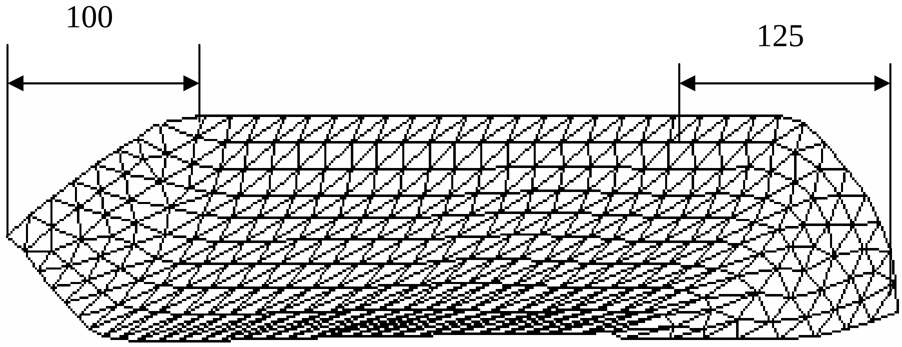
For processing of large-sized billets ∅ 200 x 100 mm, including ECAP and the subsequent upsetting, the obtained data enabled to choose the necessary length of the initial billet for ECAP, which is equal to 450 mm.

The epure analysis of normal contact stresses (Fig. 15) in the most loaded initial stage of ECAP of the billet ∅ 120 mm at 700°C indicates that in the vertical channel the stresses are distributed rather evenly and amount to about 117 MPa. Stress enhancing up to 238,8 MPa in the vertical channel and up to 253,3 MPa in the horizontal channel is observed within the area of back channel intersection angle. These die-set parts are more wearing. This fact was taken into account during designing and fabrication of the die-set. The most maximum contact stresses, acting on the puncher, amount to 322 MPa. These data are the most important for definition of pressing loads, which are estimated to 370 tons and correspondingly determine the type of pressing equipment for ECAP For the puncher, working as compressor, such loads are the critical ones and equal to about 20 % of the limit load for it.

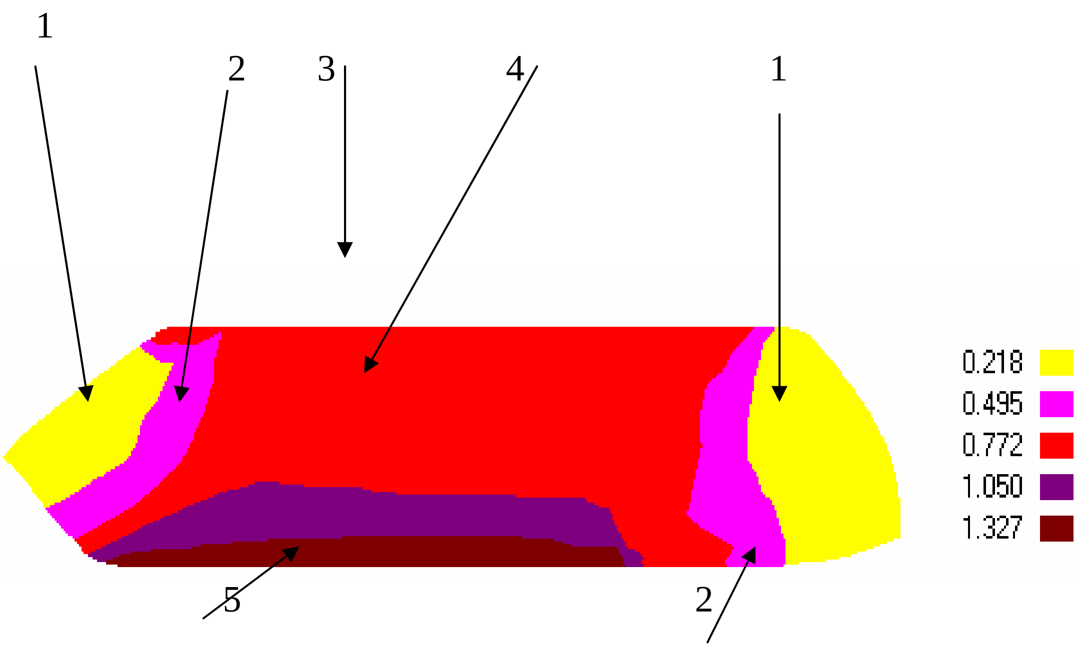
Thus, the use of computer simulation by FEM of ECAP process of the billets ∅ 120 mm allowed to minimize the expenses on the designing and fabrication of ECAP die-set and to reveal the most probable tendencies in deformation behavior of VT-6 alloy during its treatment.



a) the initial billet



b) Distorted net of finite elements



c) Distribution of accumulated strain

Fig. 14. The model of ECAP billet ∅ 120 mm, deformed at 700^0 C, $\alpha=120^0$, 1 pass. (The front part of the billet is on the right).

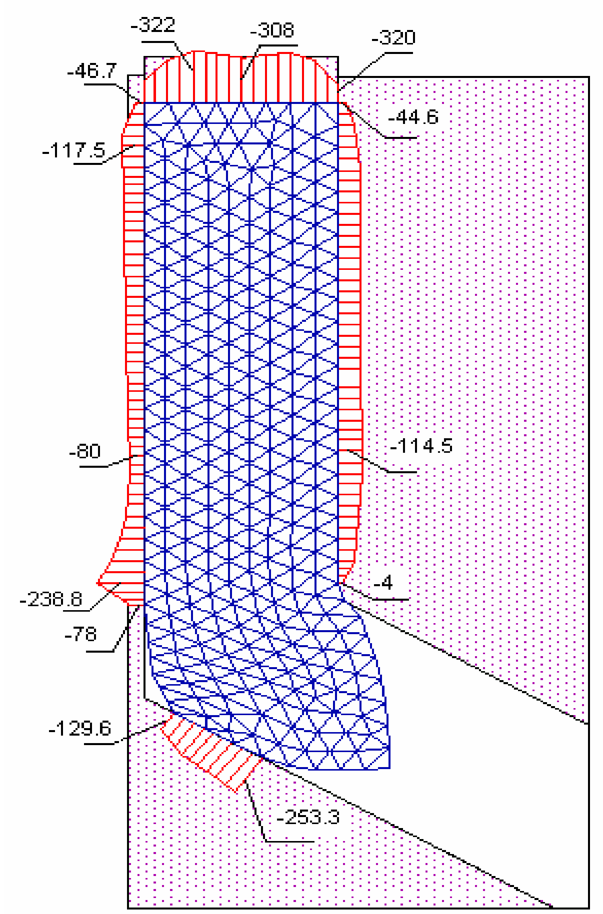


Fig. 15. The epure of normal contact stresses (MPa) for the initial stage of deformation of the billet ∅ 120 mm out of VT-6 alloy, in matrix with 120º at 700ºC.

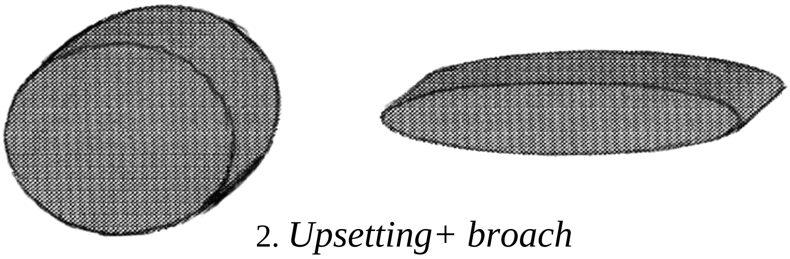
4.2. Multifold forging process

Performed investigations of ECAP of billets ∅ 20 and 40 mm have revealed important influence of the initial microstructure on the workability of VT-6 alloy. UFG state was received by using the initial billets with globular structure and grains 7-10 µm in size. The results of investigations, connected with transformation of the initial lamellar structure to the globular one in billets ∅ 40 mm, was submitted in part 3.2. It was established that ECAP at 850°C and the subsequent annealing at 800°C promote spheroidization of the initial lamellar structure and enhance workability of the alloy.

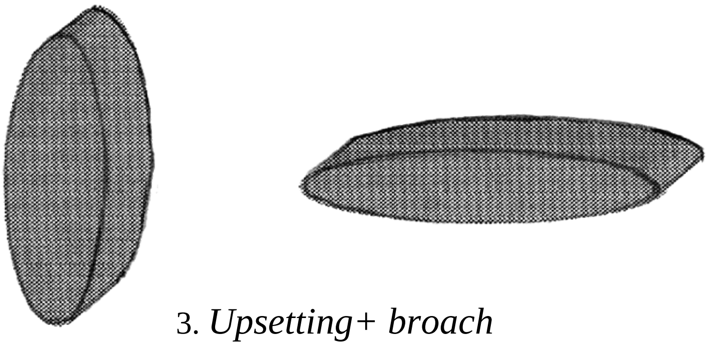
As compared to billets ∅40 mm, initial billets ∅120 mm had more coarse lamellar structure, in which the size of former β grains was about 150-300 µm. Therefore, preliminary deformation of initial billets ∅120 mm and subsequent annealing for formation globular structure was necessary. It should be taken into account that to carry out such preparation, using ECAP die-set ∅120 by analogy with formation of globular structure in billets ∅ 40 mm, was impossible. The point is that at designing stage of the die-set ECAP of billet ∅120 mm at 850°C was not planned. This die-set was not designed for pressing at such elevated temperatures. Therefore, multifold forging of the billets with changing of deformaton axis at 850°C following the scheme, submitted in Fig. 16a, and subsequent annealing at 800°C were used to refine and form globular structure and to enhance, correspondingly, workability of the alloy. The conducted treatment allowed to refine noticeably the structure in large-sized billet and to enhance potentially its workability.

Hydraulic press with loading 1600 tons (NKTB ”Iskra”, Ufa), Fig. 16b, was used to conduct this multifold forging.

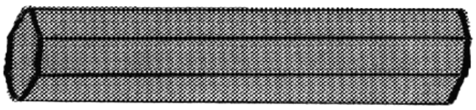
1. Upsetting



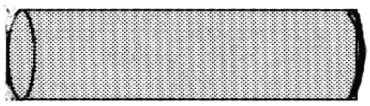
2. *Upsetting+ broach*



3. *Upsetting+ broach*

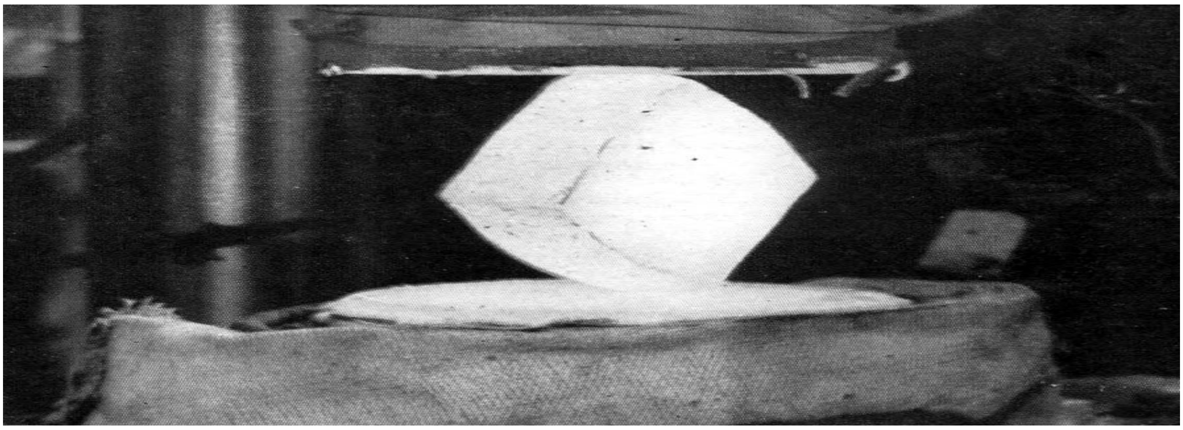


4. Roughing (cylindrical shaping



5. Machining ∅120x450 mm

a)



b)

Fig. 16. Scheme (a) and process (b) of multifold forging of the billet out of VT-6 alloy before ECAP.

4.3 Fabrication and testing of working parts of the die-set and experimental ECAP of ∅ 120 mm

One of main project tasks was die-set fabrication for ECAP of billets ∅120 mm. Basic technical parameters of experimental ECAP die-set are given below:

1. Size of initial billets, mm	∅120×450
2. Channel intersection angle	120°
3 Maximum die-set temperature, °C	450
4. Heating up to 450 °C, hour	8
5. Heater power, KWt	15
6. Working stroke, mm	550
7. Working loading, tons	700
8. Used equipment, press loading	1600

The composition and ratio of geometrical parameters of the die-set were tested during ECAP of billets ∅20 and 40 mm. On the whole the die-set composition for ECAP of billets ∅120 mm is simular to the die-set for ECAP of billets ∅40 mm (Fig. 7). It corresponds to our approach of stage ECAP scaling of billets ∅40 mm firstly, then ∅120 mm. However, double banding of insertion is important for ensuring elevated strength margin (Fig.17). At the same time the weight of such die-set amounted to 5 tons, and its fabrication costs exceed the project financing. Therefore, a die-set with only single banding was fabricated.

Using the given die-set, ECAP of billets ∅120 mm was performed. The structure of these billets was prepared following the technique, described in part 4.2. As a result in two billets one ECAP pass was performed. During the second pass of pressing in the first billet the pressing stress increased abruptly. The process was forcedly stopped. Analysis of the situation revealed that the reason was increasing of friction coefficient as a result of local adhesive gripping (welding) of the billet and die-set. It testifies to insufficient effectiveness of lubrication. The given effect was not observed during ECAP of billets ∅20 and 40 mm. It is probably connected with the scaling effect, i.e. with higher contact temperature on the boundary “billet-insertion” due to greater mass of the billet as compared to billet ∅40 mm. Several cracks occurred in the most loaded central part of insertion due to enhancing of friction and pressure forces. The given effect did not allow to proceed the following pressing as it was necessary to replace the damaged insertion with a new one.

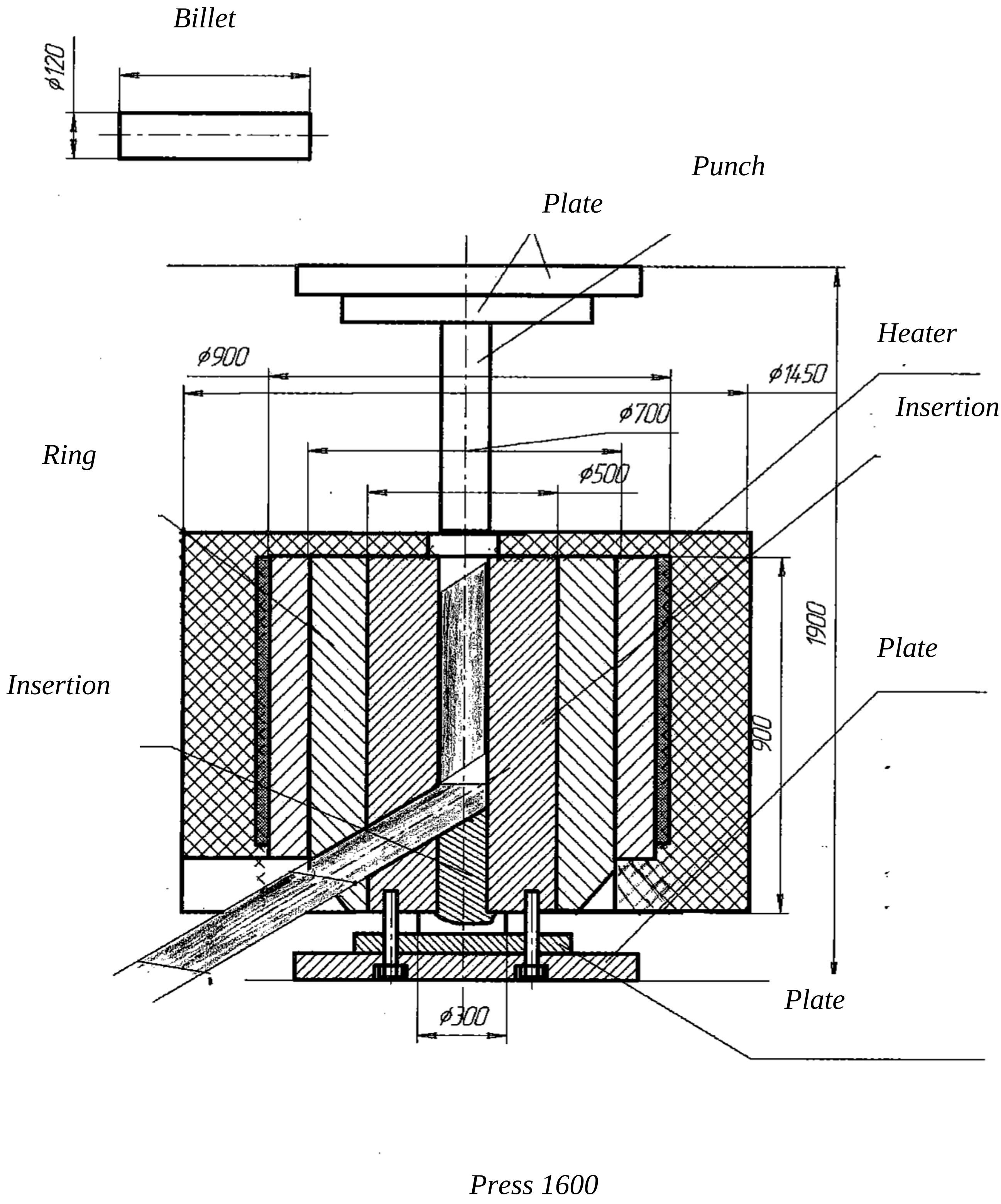


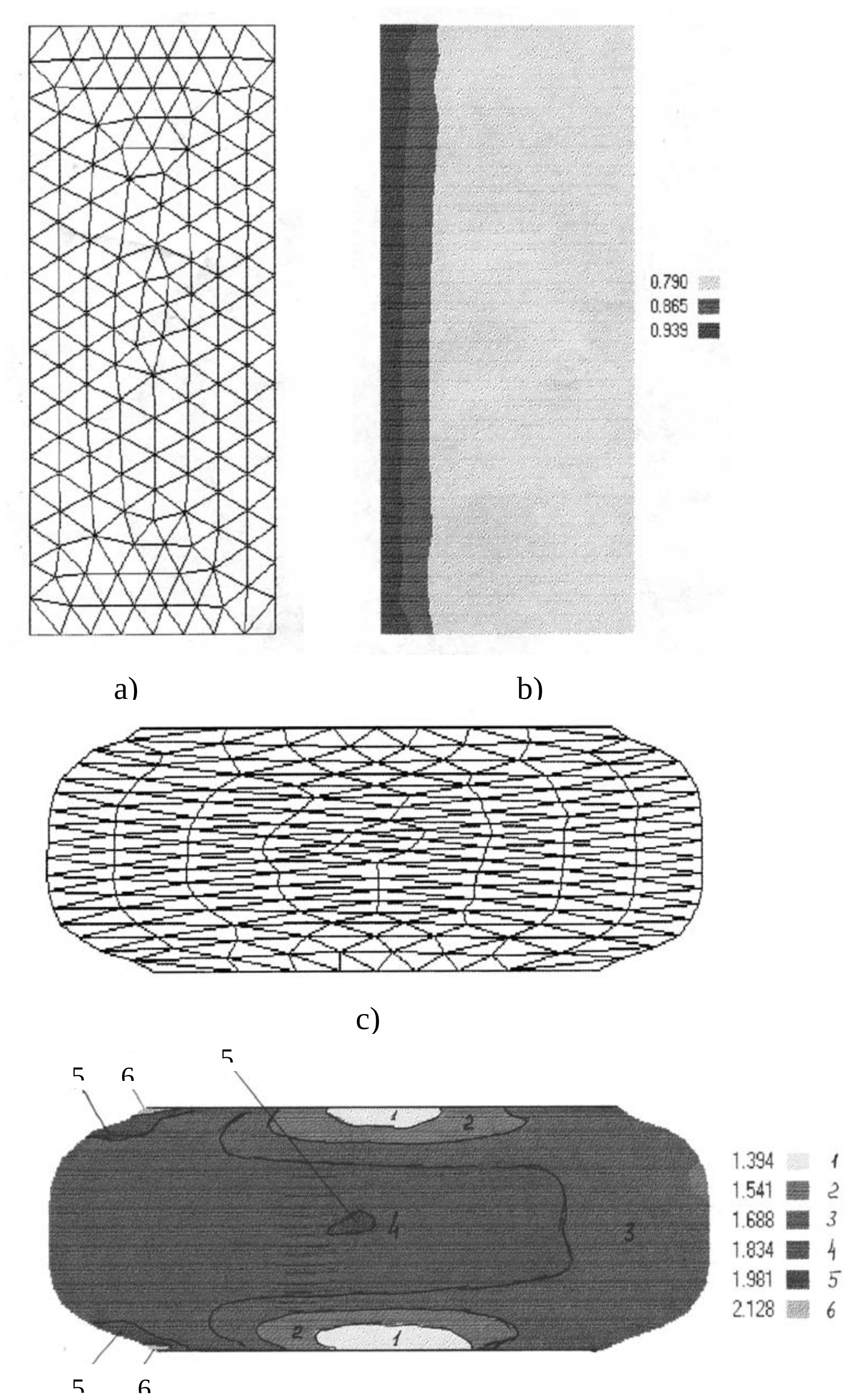
Fig. 17 Composition of ECAP die-set for billets ∅ 120 mm with double banding

4.4. Billet upsetting

As it was shown in part 4.3, we managed to perform only one pressing pass during ECAP of billet ∅ 120 mm. At that accumulated strain amounted to e = 0.7. This value is insufficient to form UFG structure in the billet. Therefore, subsequent experimental works were conducted on the given billet to shape only the final form of the forged piece.

Computer simulation of upsetting of this ECAP billet enabled to estimate quantitatively the accumulated strain in each billet's area (Fig. 19). Note should be made that distribution of finite values of accumulated strain in the forged piece is asymmetric (Fig.19). It is obviously connected with heterogeneous distribution of strain in the billet, processed after ECAP, 1pass.

To process the final form of the forged piece ∅200x100 mm we conducted singe-axis warm upsetting of ECAP billet with relative strain 60%. The initial billet before upsetting was of ∅118 x 276 mm. Small defects on the surface of the billet were mechanically eliminated. The billet was heated up to 700°C in furnace KS- 600 for two hours. The upsetting of the billet was performed at 700°C on flat blocks out of heat-resistant steel in isothermal conditions on the hydraulic press by loading of 1600 tons (Fig. 20). The maximum upsetting loading amounted to 1230 tons. After upsetting the billet possessed barrel form, that is characteristic of the pressing process. At that the maximum size of the forged piece was equal to 250 mm in diameter and 105 mm in length, and the averaged accumulated strain amounted to e = 0.85. The final form of the billet ∅200×100 mm, presented in Fig. 21, was processed by the subsequent turning.



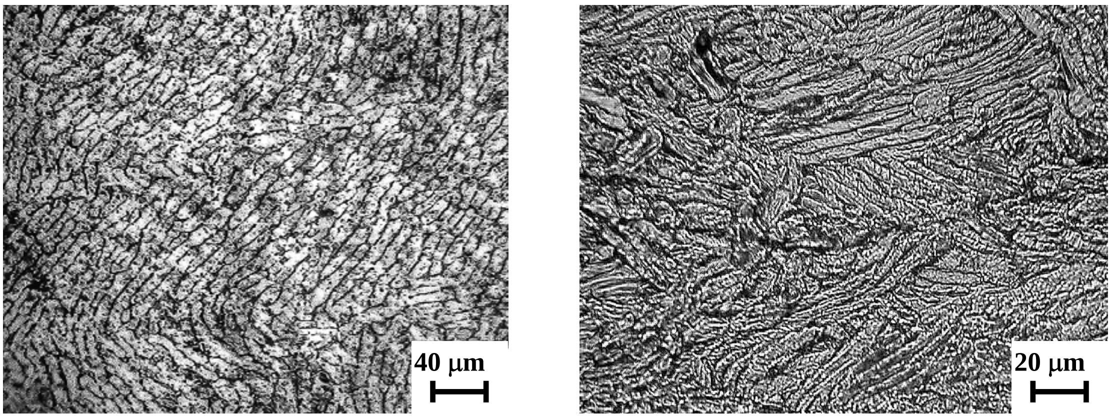
d)

Fig. 19. Pattern of distribution of accumulated strain at simulation by finite elements method of upsetting UFG billet, processed by ECAP (1 pass).

Metallographic analysis of the processed billet has shown that even after one ECAP pass and subequent warm upsetting the microstructure became more dispersed and homogeneous (Fig. 22), but for fabrication of UFG structure it is important to perform multipass ECAP.

Fig.20. Upsetting of billet out of VT 6 alloy ∅120 nm on press by loading of 1600 tons.

Fig. 21. General image of the forged piece out of VT 6 alloy after multifold forging, ECAP ∅120 mm, upsetting and machining.



(a) (b)

Fig. 22. Microstructure of the billet in the initial state (a) and after ECAP + upsetting (b) (x 500).

5. Fabrication of universal ECAP die-set with backpressure

The technical task, draft project (Fig. 23) and working drawings of the ECAP die-set for hard-deforming alloys are worked out in accordance with the statement of work (SOW). The technical task is agreed with the partner and is given below.

The technical task

The purpose: Designing and fabrication of the die-set for ECAP processing of samples at punch loading up to 1000-1400 MPa with backpressure in the horizontal pressing channel.

Basic requirements to installation:

1. Size of pressed billet, ∅ x L, mm	∅20 x 100
2. Maximal unit punch loading, MPa	1400
3. Exploitation temperature, °C	20-600
4. Maximal heat resistance of the die, °C	620
5. Short-term operation regime, no more than 30 min, °C	600
6. Long operation regime, °C	450
7. Principle of backpressure regulation	discrete
8. Principle of backpressure measurements during ECAP of tested billets	relative
9. Principle of backpressure measurements during ECAP of visco-plastic medium	direct
10. Pressure cell is installed only at the top plate of the stamp that includes a punch	
11. The die is mounted in a block with guiding columns	
12. Backpressure is created by visco-plastic medium:	
In an interval 20-300 °C	lead
In an interval 300-600 °C	aluminum, copper

The ECAP die-set was fabricated (Fig. 23), tested and dispatched to the partner.

The total weight of the die-set amounts to 200 kg. The dispatch was effected through ISTC. At present the die-set has been tested by the customer and positive conclusion has been made.

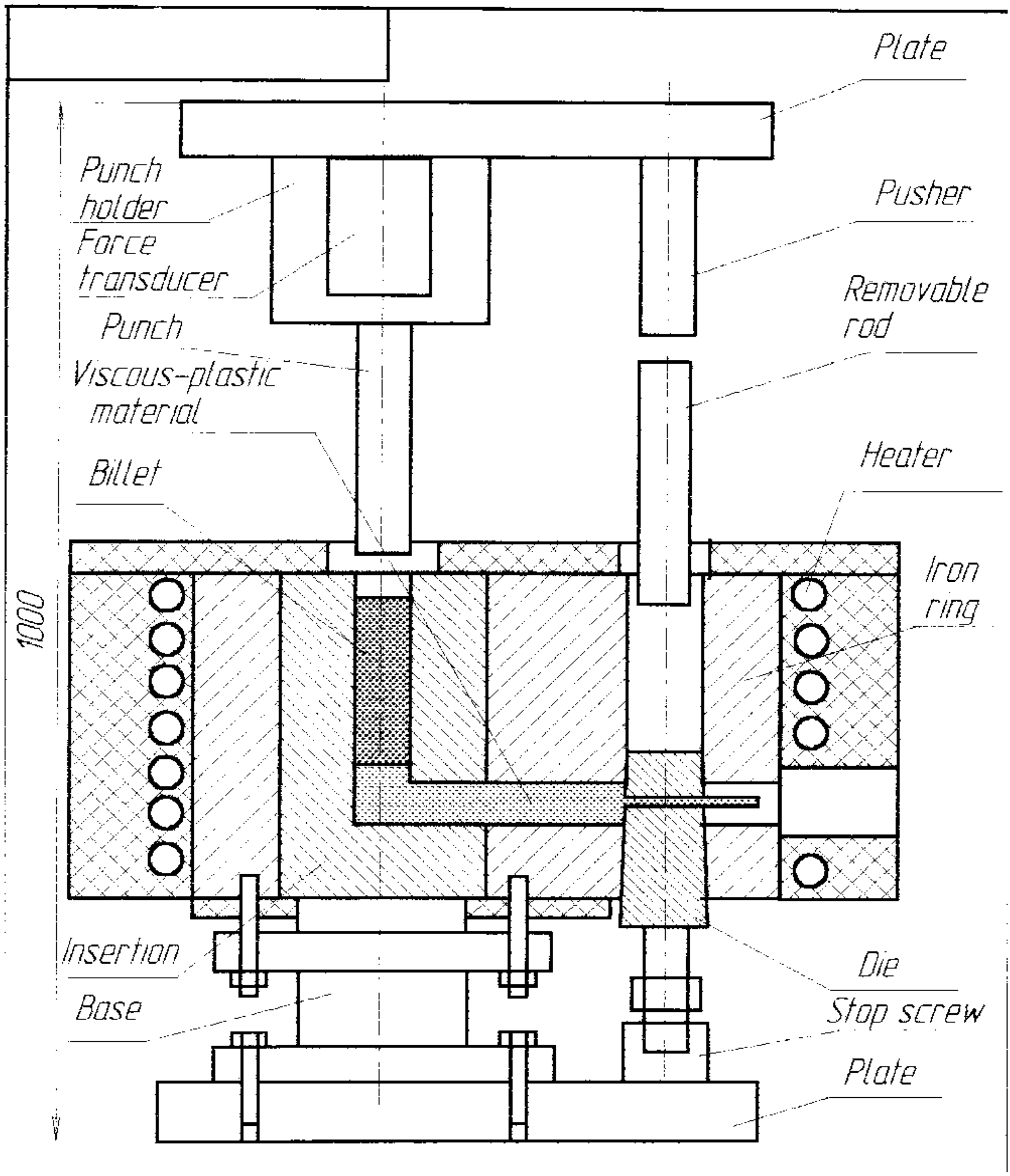


Plate
Punch holder
Force transducer
Pusher
Removable rod
Punch
Viscous-plastic material
Billet
Heater
Iron ring
1000
Insertion
Base
Die
Stop screw
Plate

a)

b)

Fig. 23. Composition (a) and a general image of main parts of the fabricated ECAP die-set for hard-to-deform alloys (b).

Conclusions

I.

Within the frames of the project processing fundamentals were developed and the possibility of ECAP processing of large-sized billets out of VT-6 (Ti-6Al-4V) alloy with ultrafine-grained structure was demonstrated.

The samples out of the alloy ∅20x80 mm with homogeneous UFG structure and the mean grain size less than 0.5 µm, were processed for the first time, using ECA pressing. The given samples after the subsequent upsetting at 600^{o}C show the extraordinary strength with ultimate tensile strength (UTS) and yield stress (YS), that amount to 1450 and 1420 MPa, correspondingly. These extraordinary values of strength are associated not only with presence of ultrafine grain of α– phase but with the structure of grain boundaries and morphology of β– phase as well.

II.

Using computer simulation and experimental study of ECAP process step-by-step approach and ECAP scaling for processing billets ∅ 40 and 120 mm with UFG structure was suggested and realized. This approach includes designing and fabrication of special ECAP die-sets, preliminary deformation treatment of the initial billets out of the alloy to transform their initial lamellar microstructures to globular ones, optimization of ECAP regimes and routes.

III.

This approach was successfully realized for processing ECAP billets ∅ 40 mm, where the alloy was preliminary subjected to ECAP at 850 ^{o}C and the subsequent annealing at 800 ^{o}C. This treatment enabled to realize spheroidization and coagulation of lamellae. It leads to formation of globular structure and enhancing of alloy workability. Then the billets were subjected to ECAP at 700 ^{o}C to process homogeneous UFG structure.

IV.

To prepare the structure of billet ∅ 120 mm multifold forging was performed. Then the billet was subjected to 1 ECAP pass and warm upsetting to process a forged piece of necessary sizes ∅ 200x100 mm. The ways of carrying out multipass ECAP were defined. However, they require designing of die-set with enhanced strength and optimization of temperature-rate ECAP regimes.

V.

The universal die-set for ECAP of billets ∅ 20 mm out of hard-to-deform alloys at 20-600^{o}C was designed, fabricated and dispatched to the partner. This die-set enables to control the stress state of the process within the changing range of hydrostatic pressure in the strain center from 500 to 1400 MPa. The given die-set was successfully tested by the customers and accepted for exploitation.

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Publication with Resume

1. V. V. Stolyarov, R. Z. Valiev, Structure-phase Transformations and Properties in Nanostructured Metastable Alloys processed by Severe Plastic Deformation, 2nd International Conference on nanomaterials by severe plastic deformation, Austria, Vienna December 9-13, 2002, will be published in a hardcover proceedings by Wiley-VCH.

In the work the potential of SPD methods for achieving metastable states is considered. There is used the recent experimental results as well as the comparison of some structure feature and mechanical properties of several Ti and Al based alloys, subjected to two SPD methods which are presently the most widely used, namely, high pressure torsion (HPT) and equal channel angular pressing (ECAP).

List of Conference reports with Resumes

1. V. V. Stolyarov, R. Z. Valiev, Bulk nanostructured alloys from severe plastic deformation, International Workshop “New Production Technologies and Materials”, Portugal, Lisbon, December 2-5, 2002.

Recent results of the development of the severe plastic deformation method namely equal channel angular pressing (ECAP) to fabricate bulk nanostructured alloys as well as results of their thorough structural characterization and investigations of their unusual deformation behavior and novel mechanical properties are overviewed and discussed.

2. V. V. Stolyarov, R. Z. Valiev, Structure-phase Transformations and Properties in Nanostructured Metastable Alloys processed by Severe Plastic Deformation, 2nd International Conference on nanomaterials by severe plastic deformation, Austria, Vienna December 9-13, 2002.

In the work the potential of SPD methods for achieving metastable states is considered. There is used the recent experimental results as well as the comparison of some structure feature and mechanical properties of several Ti and Al based alloys, subjected to two SPD methods which are presently the most widely used, namely, high pressure torsion (HPT) and equal channel angular pressing (ECAP).

Patent and Author’s Rights Information

1) Russian patent № 2181314. Apparatus for treatment of metals by high deformation and pressure / Raab G.I., Kulyasov G.V., Polozovsky V.A., Valiev R.Z.- Published on 20.04.2002.

The know-how is aimed at improvement of device construction for ECAP of materials. The features of constructive fulfillment of pressing channels, aimed at enhancing deformation homogeneity and decreasing pressing loads, are reflected in it.

ISTC 2125p

Final
Project Technical Report
of ISTC 2125p

Fabrication and investigations of large-sized billets out of Ti-6Al-4V alloy with ultrafine-grained structure using SPD-processing

II. Project Technical Report

(From 1 September 2001 to 28 February 2003 for 18 months)

Ruslan Zufarovich Valiev
(Project Manager)
Ufa State Aviation Technical University

March 2003

Vice-rector USATU, Professor **V.S. Zhernakov**

Project manager, Professor **R.Z.Valiev**

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The objective of this project is to develop equal channel angular pressing (ECAP), as method of severe plastic deformation (SPD), for fabrication and investigation of large-sized billets out of VT 6 (Ti-6Al-4V) alloy, containing ultrafine-grained (UFG) structure with grain size less than 0.5 μm).

Project summary: Within the frames of the project complex investigations, aimed at processing of large-sized billets out of VT 6 (Ti-6Al-4V) alloy with UFG structure, have been performed.

Formation of homogeneous UFG structure with grain size less than 0.4 μm in billets ∅ 20 mm by ECAP technique and subsequent warm upsetting has been shown. It has been demonstrated that such treatment results in considerable increase of strength properties of the alloy. Their values reach ultimate tensile strength of 1450 MPa and yield stress of 1420 MPa.

Using computer and experimental simulation of ECAP process, the possibility of its scaling for fabrication of large-sized billets of ∅ 40 and 120 mm and the subsequent upsetting has been developed.

Keywords: titanium alloy Ti-6Al-4V (VT6), ultrafine-grained structure, large-sized billets, severe plastic deformation, equal channel angular pressing.

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INTRODUCTION

The present final report represents the results of investigations that demonstrate for the first time the possibility of using equal channel angular pressing (ECAP) to process large-sized Ti-6Al-4V billets with ultrafine grain size (UFG) less than 0.5 μm.

In recent years ECAP arises a great interest as the method of severe plastic deformation (SPD) that allows to form UFG structure in different metals and alloys and, as a result, to enhance their mechanical properties [1-5]. However, its application to hard-to-deform materials, where titanium alloys belong, requires special investigations connected with increase of their workability, ECAP process simulation, study of strain localization. The task of ECAP scaling to increase the sizes of processed billets is particularly important for practical application. Some of these investigations were carried out for ECAP Ti-6Al-4V alloy as well [4, 5, 6]. For example, within the range of relatively high deformation temperature 845-955 °C a fine-grained structure with grain size 10 μm was processed by ECAP [5]. The investigation results of the influence of initial structure in samples ∅9.5 mm out of Ti-6Al-4V alloy on structure evolution during ECAP at temperature 500-700 °C with the maximum number of passes 4 have been recently published as well [6]. Nevertheless, the problem of processing bulk billets ∅ 20 mm and more with UFG structure out of Ti-6Al-4V alloy by ECAP still remains highly topical and that is connected with great complexity of carrying out severe deformation of the given alloy. This task requires integrated approach and overcoming of many problems related to materials science and forming for hard to deform materials, that have been investigated within the frames of the present project:

a) Numerical simulation and experimental studies of ECAP of the alloy.
b) Designing and fabrication of the ECAP die-sets for processing billets ∅ 20 mm, ∅ 40 and ∅120 mm. Optimization of ECAP regimes using the results of the experiments.
c) ECAP processing the given billets with UFG structure and then their upsetting to fabricate the billets in the form of disks.
d) Investigation of microstructure homogeneity, phase composition and mechanical properties in the processed large-sized billets.

The results on designing and fabricating of ECAP die-sets, ECAP modeling, optimization of ECAP regimes as well as the investigation results of microstructure and mechanical properties of processed samples ∅ 20 mm, ∅40mm and ∅120mm out of VT-6 alloy, the Russian analogue of Ti-6Al-4V alloy (ASTMF 136), are presented in the given report.

The final project goal was processing large-sized billet ∅200x100 mm with UFG structure, that supposed ECAP of billets ∅120x450 mm, subsequent machining up to ∅120x220 mm and then its warm upsetting up to ∅200x100 mm.

1. Initial material and experimental procedures

In the work the VT-6 alloy was used that is a Russian analogue of the commercial Ti-6Al-4V alloy (ASTM F 136). The alloy was manufactured and supplied by Verkhne-Saldinsky metallurgical production association (AO VSMPO) in the form of hot-rolled bars ∅ 25 mm, ∅ 40 mm and 150 mm. The temperature of polymorphous $\alpha \rightarrow \beta$ transformation is 990°C. The chemical composition of the alloy is given in Table 1.

Table 1. Chemical composition of VT6 in weight % (actual and in accordance with the standard)

	Ti	**V**	**Al**	**C**	**N**	**Fe**	**Si**	**H**	**O**
GOST	Base	3.5…5.3	5.3…6.8	0.10	0.05	0.60	0.10	0.015	0.20
Act.	Base	4.8	6.6	0.02	0.01	0.13	0.04	0.002	

The initial microstructure was globular for rods ∅25 mm (Fig.1a) and lamellar for rods ∅40 and 150 mm (Fig.1b).

a)

b)

Fig. 1. Optical microphotography. The structure of the initial alloy: a) globular; b) lamellar

Various character of microstructure in the initial billets of the alloy is connected with the difference of thermo- mechanical conditions of hot rolling of rods of various diameter, in particular, with differences of temperature during the final rolling stage and cooling rates.

The structure investigations were carried out using the optical metallography and the transmission electron microscopy methods.

For the analysis of the structure uniformity the microhardness was measured.

The tensile tests were carried out at room temperature using standard cylindrical samples ∅5x25 mm, cut out in the direction of the pressing axis, or flat microsamples 5x0.7x2.5 mm, cut out transversely to the upsetting direction. For two types of samples the tensile rate amounted to 0,5 mm/min.

2. Processing and investigation of ECAP samples ∅ 20 mm

Taking into account the novelty and complexity of the task put by, experimental investigations of ECAP samples ∅20 mm have been carried out at the first stage.

The results of the investigations are given below, they included simulation of ECAP process, the experiments on workability of the billets in various structural states during ECAP, and also structural characterization, microhardness measurements and tensile tests, aimed at definition of regimes, forming a homogeneous UFG structure in the samples out of VT6 alloy.

2.1. Computer simulation by finite elements modeling (FEM)

The main goal of this simulation was the investigation of workability of the alloy samples during multipass pressing. It is known [7], that for pressing route Bc, in which the billet revolves by 90° on its longitudinal axis between pressing passes, to form UFG structure it is necessary to attain the level of accumulated strain higher than e= 4-5.Therefore, the main task for UFG structure formation was to secure the level of accumulated strain equal to e= 5-6 without failure of the billets. The approach [8], including the analysis of deformability reserve on the basis of plasticity diagram of the alloy within the coordinates ($\sigma-\Lambda$), where stress σ and degree of shear strain Λ are the parameters, characterizing stress-strain state of the process. This approach enables to reveal values of σ and Λ, at which the first crack can appear. To build the plasticity diagram experimental data on single axis upsetting and torsion of the alloy at 700°C were used. In accordance with the data obtained, the strain of the alloy should not exceed e =0,8- 0,9 during each pass of ECAP.

Computer simulation of ECAP billets ∅20 mm was performed at channel intersection angle 90, 110, 120, 135°. It was established that for pressing temperature 700°C, angles 120° and 135° the combination of plasticity reserve and strain, accumulated per one pass, can secure processing of integral samples without failure. The image of distribution of accumulated strain per one pass for channel intersection angle 120° is given in Fig. 2. Numeric values of deformation are defined by simulation for the part of the billet, corresponding to the established stage of material flow. At that the defined value range e =0,55-0,65 does not exceed the ultimate values of workability e=0,8- 0,9 in accordance with plasticity diagram. The analysis of homogeneity of deformation distribution in the sample shows that the front part of the billet is the most heterogeneous. Within 30 mm from the end of the billet the accumulated strain changes from 0 to 0,59. This fact should be taken into account for studying structure and mechanical properties of billet material after ECAP. The values of accumulated strain within the area of top surface, the most dangerous from the viewpoint of appearance and development of cracks, are equal to e=0,59-0,71. They are lower as well than the ultimate values. The maximum values of accumulated strain e=0,82…0,94 have been revealed in the area that adjoins to the lower angle of channel intersection. The given level corresponds to the ultimate values of accumulated strain. However, the cases of appearance of cracks in the lower surface of billets are not known in practice.

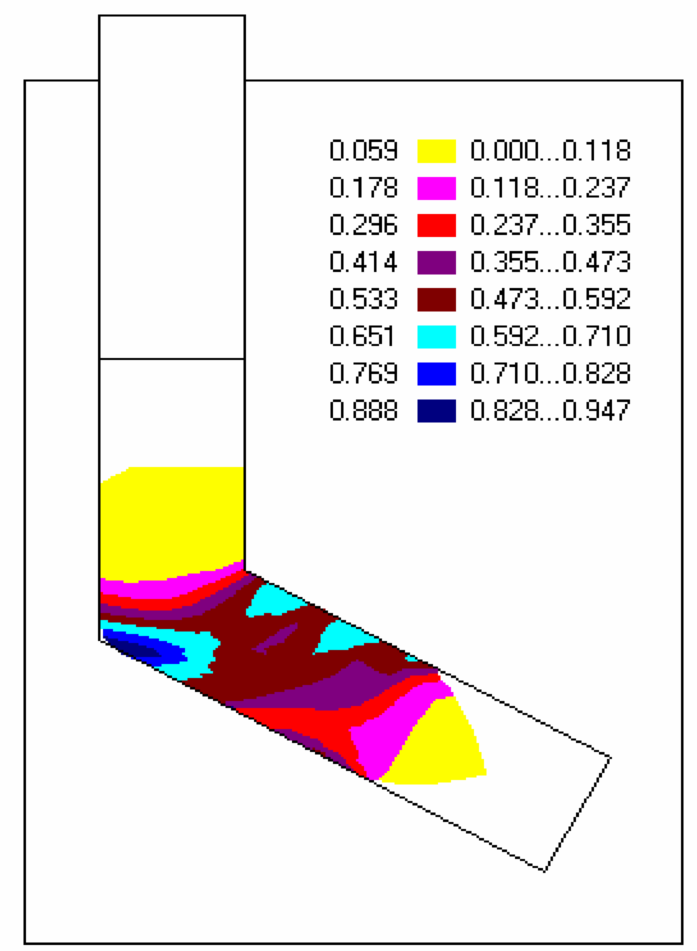


Fig. 2. Distribution of accumulated strains in the sample ∅ 20 mm out of VT-6 alloy during ECAP processing at 700 ^{0}C, α=120^0, pass 1.

2.2. ECAP processing in various regimes

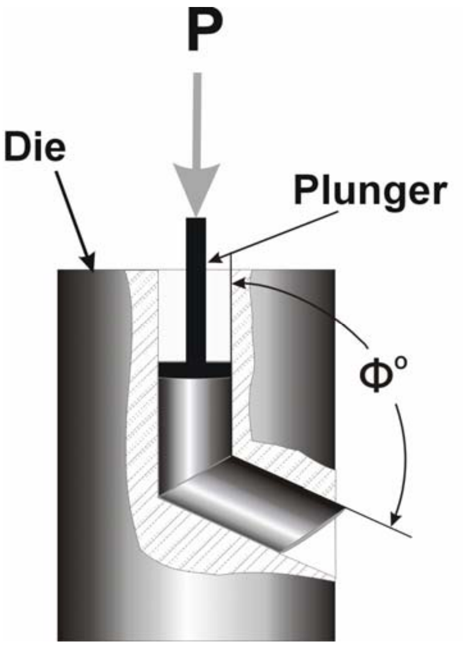
ECAP was carried out with billets ∅ 20 x 70 mm in three structural states:

1) as received state;
2) quenched from (α+β) area at 950°C;
3) quenched from β area at 1010°C.

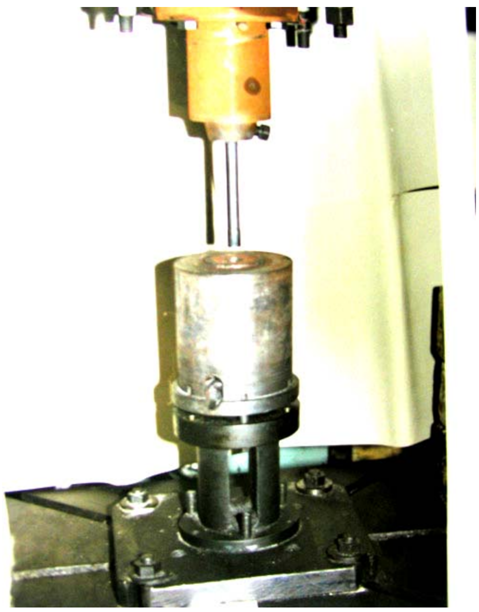
ECAP was realized using the die, which scheme is shown in Fig. 3 b, which was designed by us earlier and was used for ECAP of commercially pure titanium [9]. The scheme of the process is given in Fig. 3 a.

Pressing parameters:

Billet temperature, °C	700 and 800
Channel intersection angle, °	135
Pressing route	B_c
Pressing rate, mm/sec	6
Lubrication	"Angelina" (the stuff - graphite)



a)



b)

Fig. 3. Principle ECAP scheme (a) and die-set (b) for ECAP samples of the alloy ∅20 mm

Before ECAP the billets were heated up to the chosen temperature in a furnace for 15 min, then they were replaced into the ECAP die-set, heated up to 500°C and were further deformed

The minimum (700°C) and the maximum (800°C) temperature of the billet was determined upon the sufficient alloy workability and thermal stability of the deformed alloy.

An important parameter of the ECAP process is a channel intersection optimal angle, found through the experiment, equal to 135°, which enables to reach the needed accumulated strain in billets without their failure.

ECAP of billets was carried out in route Bc. The workability investigation of the billets, quenched from α+β or β areas, demonstrated that these states due to formation of low plastic martensite structure had a negative influence on alloy workability. In this connection the results of investigations of quenched states are not presented in the given report.

2.3. Structure characterization and microhardness measurements

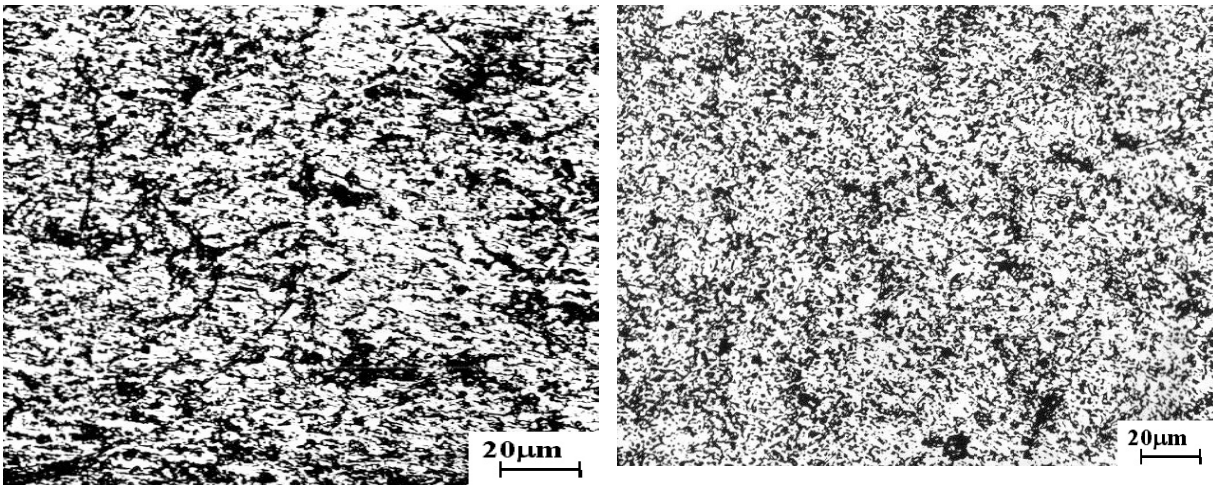
The aim of this research is the study of structure uniformity in the volume of the deformed samples, and also morphology and size of phases, formed during ECAP process for various conditions.

Optical metallography

The initial state of rods ∅25 mm is characterized by the fine-grained globular structure mainly (Fig. 1a). The structure peculiarity is the presence of rather large globules up to 6 μm of the primary β - phase and plate-like dispersed precipitation of the secondary α - phase.

Multipass ECAP with 13 passes at billet temperature 700°C and channel angle 135° causes a notable refinement of the initial structure in the cross section (Fig. 4, a) up to grain size about 0.5 μm. The slight structure elongation in the direction of the rolling typical for the initial alloy practically disappears. Metallographic analysis, performed in three arbitrary cross sections along the rod's length, has shown high structure homogeneity of deformed samples in longitudinal and cross sections.

The billet temperature increase from 700 °C up to 800 °C had practically no influence on the microstructure that is observed through an optical microscope (Fig. 4.b).



a) b)

Fig. 4. Optical metallography. The initial alloy microstructure in the cross section after ECAP at billet temperature 700°C – 13 passes (a) and 800°C – 16 passes (b).

Microhardness

The results of microhardness measurements for various states of VT6 alloy in the longitudinal and the cross sections of the template cut out of the central part of the bar are demonstrated in Table. 2.

Table 2. Microhardness of VT6 alloy in various states

#	State	Microhardness, MPa	
		Longitudinal section	Cross section
1.	Initial	2810±50	3020±70
2.	1 + ECAP (700°C – 13 passes)	4240±100	4000±120
3.	2+ upsetting (600°C-55%)	-	4050±120

As it is seen from Table 2, ECAP billet at 700 °C results in microhardness growth in 1.5 times as compared to the initial state. A deformed bar in the longitudinal section has a little higher microhardness than a bar in the cross section. It testifies to absence of any noticeable anisotropy after ECAP. Additional upsetting of ECAP samples does not influence on microhardness in the cross section, the value of which is equal to microhardness of ECAP sample within accuracy range. Nevertheless, strength tensile properties change considerably (see Table 3 below).

TEM observations

Structure TEM photographs of the samples processed by ECAP at billet temperatures 700 and 800 °C are described in Fig. 5 (a, b).

As it is seen, after ECAP at the billet temperature of 700 °C the SAED pattern taken off from the areas of 2 μm^2 looks like rings with the numerous spots, randomly distributed on them. Such electron-diffraction pattern can testify to ultrafine-grained structure formation where high-angle boundaries are present. The bright-field image testifies to the grain size less 0.5 μm. The microstructure is characterized by high density of various defects (dislocations, grain/subgrain boundaries) and high internal stresses.

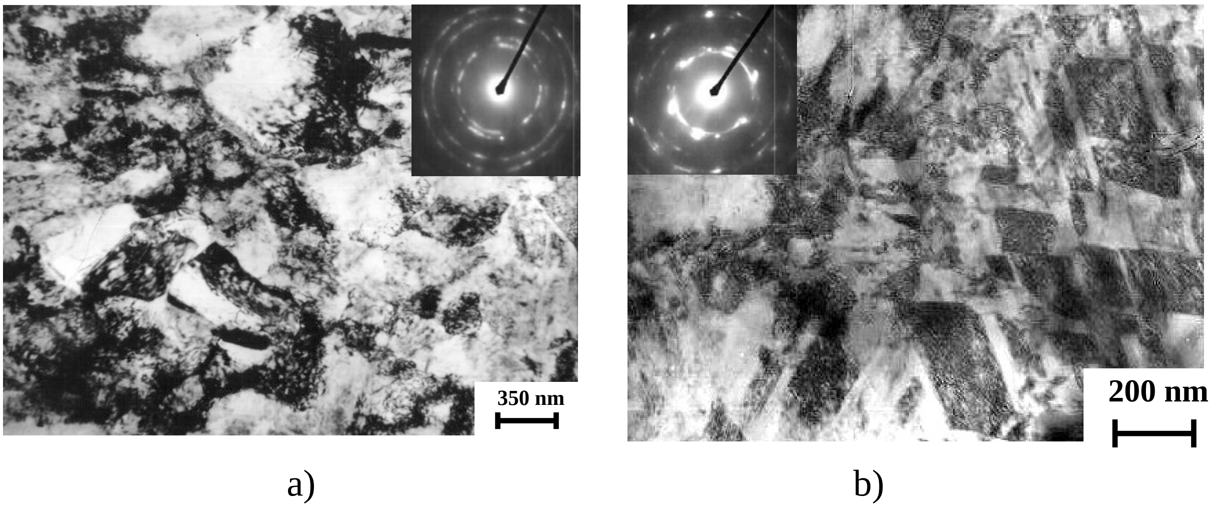


Fig. 5. TEM and SAED pattern in the cross section of ECAP VT6 alloy, deformed at billet temperature a) 700°C - 13 passes; b) 800°C – 16 passes.

Microstructure and the SAED pattern for the ECAP state at billet temperature 800°C appreciably differ from those for an ECAP alloy at 700°C (Fig.5, b). Increase of α - plates volume fraction and length takes place. This fact is in accordance with the equilibrium phase diagram and reflects a change of quantitative ratio of α / β phases and growth of transformed β – phase grains at higher temperature.

The morphology of structural elements is mainly plate-like, the plates 100 nm thick and 400-500 nm long. However, in some areas of the sample there are areas with grain structure.

2.4. Mechanical properties

The results of tensile tests of VT6 alloy in various states, are presented in Table 3. The results testify that ECAP for all investigated states leads to increasing of strength characteristics of UTS and YS by 20 % in comparison with the initial state of the alloy. At this the elongation before failure though decreases, but it remains high enough.

Comparison of mechanical properties and the literary data for Ti-6Al-4V and VT6 alloys, subjected to traditional methods of strengthening processing (high-temperature forging [10], rolling [11]), shows an appreciable advantage of ECAP method for receiving more high-strength states. Higher UTS 1300 MPa in a similar alloy was obtained by the multifold isothermal forging method [12], however the size of the processed billet was only ∅ 10 x 15 mm. With greater size of billets the UTS of a VT6 alloy, processed by the same method, did not exceed 994 MPa [13].

Table 3. Mechanical properties of VT6 alloy in various states

#	State	UTS, MPa	YS, MPa	δ, %
1	Initial (as received)	970	900	20
2	1 + ECAP at 700 °C, 13 passes	1160	1110	12
3	2 + upsetting at 650 °C – 1 hour	1140	1100	8
4	2 + upsetting at 600 °C, ε = 55%)	1450*	1420*	11*

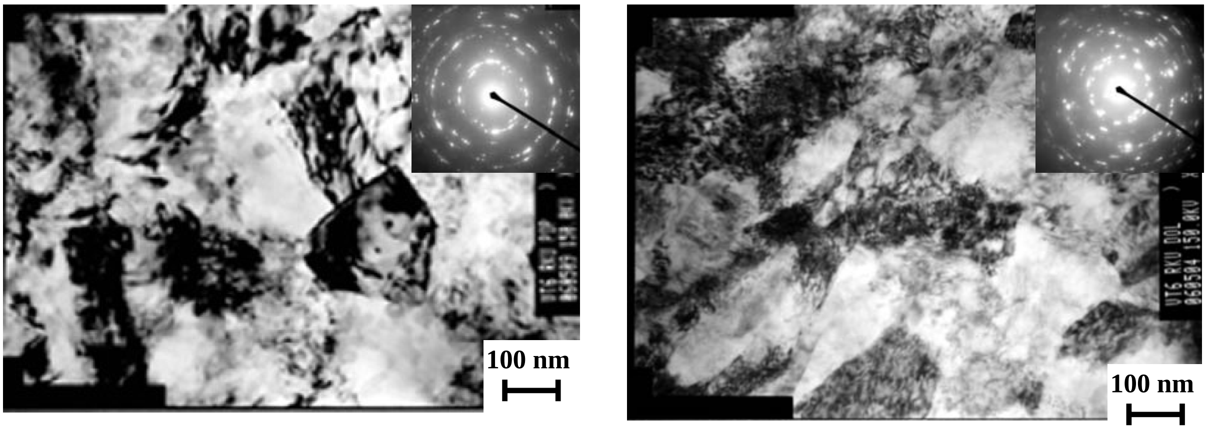
for states 1-3 the samples ∅ 5 x 25 mm were tested;
* for state 4 the samples 5 x 7 x 2.5 mm were tested.

2.5 Upsetting of ECAP billet

Upsetting is a responsible forming operation. At this stage it is necessary to exclude defects in the form of chips and fractures on the billet surface. Taking into account the fact that during upsetting it is necessary to secure high strain degree (more than 50 %) at relative low temperatures (no more than 700°C), the indicated task is viewed as a rather complex one. Therefore, the initial upsetting experiments were carried out on the modeling billet ∅ 20 x 22 mm. The goal of the work was a search of optimal temperature- rate conditions of the upsetting and investigation of its influence on mechanical properties and microstructure of the deformed billet.

VT-6 alloy after ECAP at 700°C with strain degree, 13 passes used as initial state before upsetting. To decrease friction factor the turning 0,3 mm deep was made on face planes of the billet. Before upsetting this turning was filled up with graphite lubrication. Such technique secured more homogeneous deformation along the height of the billet during the upsetting process. The upsetting was performed at 600 °C by pressing with the average strain rate 10^{-3} s^{-1}. Taking into account the small grain size, it enabled to secure the conditions close to superplastic deformation [14].

The results of microstructure investigations after ECAP and additional upsetting at 600°C with strain degree 55 % have shown that a mean grain size even decreases slightly as compared to ECAP state and amounts to 0,4 µm. The grain form is equiaxed mainly both in longitudinal and cross sections with regard to the upsetting direction. (Fig. 6). The image of electron-diffraction patterns with many spots along circles testifies that the forming structure is the structure of grain type with high-angle misorientations. The observed spreading of spots in electron-diffraction patterns testifies the presence of internal stresses. Lattice dislocations are obviously considered to be the source of these stresses.



a) b)

Fig. 6. Microstructure of the VT-6 alloy, subjected to ECAP at 700°C - 13 passes and upsetting at 600 °C, ε = 55%: a) cross section, b) longitudinal section.

The results of mechanical tests of ECAP alloy after upsetting are presented in Table 3. Earlier conducted comparative investigations on the samples of different forms and geometry have shown that, as a rule, the strength characteristics are lower, and elongation is higher for flat micro- samples as compared to standard cylindrical ones. In this connection it is correctly to carry out the comparison of only strength characteristics. The data of Table 3 show that UTS of ECAP alloy after upsetting is higher by 27 % than the initial ECAP. It seems connected with the formation of more equiaxed structure with smaller grain size. The achieved UTS (1450 MPa) approaches to record level for such type of titanium alloys. Nevertheless, this value is less than the ultimate tensile strength 1750 MPa obtained in [15]. However, this strength was achieved by the other SPD method –high pressure torsion on the small samples.

ISTC 2125p

Final
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Fabrication and investigations of large-sized billets out of Ti-6Al-4V alloy with ultrafine-grained structure using SPD-processing

III. Project Summary

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The objective of this project is to develop equal channel angular pressing (ECAP), as method of severe plastic deformation (SPD), for fabrication and investigation of large-sized billets out of VT 6 (Ti-6Al-4V) alloy, containing ultrafine-grained (UFG) structure with grain size less than 0.5 μm).

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Formation of homogeneous UFG structure with grain size less than 0.4 μm in billets ∅ 20 mm by ECAP technique and subsequent warm upsetting has been shown. It has been demonstrated that such treatment results in considerable increase of strength properties of the alloy. Their values reach ultimate tensile strength of 1450 MPa and yield stress of 1420 MPa.

Using computer and experimental simulation of ECAP process, the possibility of its scaling for fabrication of large-sized billets of ∅ 40 and 120 mm and the subsequent upsetting has been developed.

Keywords: titanium alloy Ti-6Al-4V (VT6), ultrafine-grained structure, large-sized billets, severe plastic deformation, equal channel angular pressing.

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Brief Project Summary

The objective of the given project is to develop equal channel angular pressing (ECAP), as method of severe plastic deformation (SPD) for fabrication and investigation of large-sized billets out of VT 6 (Ti-6Al-4V) alloy containing UFG structures (with grain size less than 0.5 µm).

In recent years ECAP arises a great interest as the method of severe plastic deformation (SPD) that allows to form UFG structure in different metals and alloys and, as a result, to enhance their mechanical properties. However, its application to hard-to-deform materials, where titanium alloys belong, requires special investigations connected with increase of their workability, ECAP process simulation, study of strain localization. The task of ECAP scaling to increase the sizes of processed billets is particularly important for practical application. The problem of processing bulk billets ∅ 20 mm and more with UFG structure out of Ti-6Al-4V alloy by ECAP still remains highly topical and that is connected with great complexity of carrying out severe deformation of the given alloy. This problem required integrated approach and overcoming of many problems related to materials science and forming for hard to deform materials, that have been investigated within the frames of the present project:

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The final project goal was processing large-sized billet ∅200x100 mm with UFG structure, that supposed ECAP of billets ∅120x450 mm, subsequent machining up to ∅120x220 mm and then its warm upsetting up to ∅200x100 mm.

Within the frames of the project scientific fundamentals were established and the possibility of ECAP processing of large-sized billets out of VT-6 (Ti-6Al-4V) alloy with ultrafine-grained structure was demonstrated.

The samples out of the alloy ∅20x80 mm with homogeneous UFG structure and the mean grain size less than 0.5 μm, were processed for the first time, using ECA pressing. The given samples after the subsequent upsetting at 600°C show the very important strength with ultimate tensile strength (UTS) and yield stress (YS), that amount to 1450 and 1420 MPa, correspondingly.

Using computer simulation and experimental study of ECAP process step-by-step approach and ECAP scaling for processing billets ∅ 40 and 120 mm with UFG structure was suggested and realized. This approach includes designing and fabrication of special ECAP die-sets, preliminary deformation treatment of the initial billets out of the alloy to transform their initial lamellar microstructures to globular ones, optimization of ECAP regimes and routes.

This approach was successfully realized for processing ECAP billets ∅ 40 mm, where the structure was preliminary subjected to ECAP at 850 °C and the subsequent annealing at 800 °C. Then the billets were subjected to ECAP at 700 °C to process homogeneous UFG structure.

To prepare the structure of billet ∅ 120 mm multifold forging was. Then the billet was subjected to 1 ECAP pass and warm upsetting to process a forged piece of necessary sizes ∅ 200x100 mm. The ways of carrying out multipass ECAP were defined. However, they require designing of die-set with enhanced strength and optimization of temperature-rate ECAP regimes.

The universal die-set for ECAP of billets ∅ 20 mm out of hard-to-deform alloys at 20-600°C was designed, fabricated and dispatched to the partner. This die-set enables to control the stress state of the process within the changing range of hydrostatic pressure in the strain center from 500 to 1400 MPa. The given die-set was successfully tested by the customers and accepted for exploitation.

1. List of publications

1. V. V. Stolyarov, R. Z. Valiev, Structure-phase Transformations and Properties in Nanostructured Metastable Alloys processed by Severe Plastic Deformation, 2nd International Conference on nanomaterials by severe plastic deformation, Austria, Vienna December 9-13, 2002, will be published in a hardcover proceedings by Wiley-VCH.

2. V. V. Stolyarov, R. Z. Valiev, Bulk nanostructured alloys from severe plastic deformation, International Workshop "New Production Technologies and Materials", Portugal, Lisbon, December 2-5, 2002.

3. V. V. Stolyarov, R. Z. Valiev, Structure-phase Transformations and Properties in Nanostructured Metastable Alloys processed by Severe Plastic Deformation, 2nd International Conference on nanomaterials by severe plastic deformation, Austria, Vienna December 9-13, 2002.

1) Russian patent № 2181314. Apparatus for treatment of metals by high deformation and pressure / Raab G.I., Kulyasov G.V., Polozovsky V.A., Valiev R.Z.- Published on 20.04.2002.

The know-how is aimed at improvement of device construction for ECAP of materials. The features of constructive fulfillment of pressing canals, aimed at enhancing deformation homogeneity and decreasing pressing loads, are reflected in it.

Technology Implementation Plan

What marketable results have been achieved or are anticipated?

№	Title of the result possible for utilization	Type of intellectual property	Owner of the results	Intention for utilization	Notes
1	Ultrafine- grained (UFG) large-sized billets out of VT 6 with enhanced static and cyclic strength	none	none	none	Partnering application for patent “Processing technique for large-sized UFG billets out of Ti-6Al-4V alloy”
2	Apparatus for treatment of metals by pressure / Raab G.I., Kulyasov G.V., Polozovsky V.A., Valiev R.Z.	Russian patent № 2181314. Published on 20.04.2002.	Russia	As pilot samples prototypes of devices for processing UFG titanium billets	-

Describe the current Stage of Development of the project result:

Development stage	**Results (numbered 1-4 per table above point 1)**
Basic Research	Further investigations of transformation of lamellar structure to globular one in large-sized billets
Applied Research Optimization / Prototype manufacture	Optimization of ECAP regimes for processing large-sized billets with UFG structure. Designing of scientific technological die-set and its fabrication. Processing of pilot billets, characterization of their structure and mechanical properties
Other	

What is the status of Intellectual Property Rights to your Results?

Type of the rights on IP	Territories, numbers of patent applications and patents	Background or Foreground Rights
Partnering application for patent “Processing technique for large-sized UFG billets out of Ti-6Al-4V alloy” will be submitted	Russia, USA	-
Positive decision on the application	-	-
Patent is received	Russian patent № 2181314. Published on 20.04.2002.	Prior intellectual property rights belong to USATU
Utility model is registered	-	-
Copyright	-	-
Know-how	-	Legalization of rights, that belong to the leading project participants, is planned, know-how including
Other	-	-

Summarize approximate schedule for activities planned to implement your project results and "graduate" your project team:

Activity on introduction of results	**Attracted partners**	**Work schedule (starting from…to …)**	**Evaluation of estimated expenses**
Fundamental investigations of preliminary preparation of structure of large-sized billets for ECAP. Optimization of ECAP regimes for processing large-sized billets with UFG structure. Designing of scientific technological die-set and its fabrication. Processing of pilot billets, characterization of their structure and mechanical properties.	EOARD	From 2004 to 2005	$120,000

What is your Plan to reach your target industry sector / public-sector organization with your message of sponsorship of your result?

Conference(s)

1. 2nd International Conference on nanomaterials by severe plastic deformation, Austria, Vienna December 9-13, 2002.

2. International Workshop "New Production Technologies and Materials", Portugal, Lisbon, December 2-5, 2002

Collaborator

The work was performed in cooperation with the representative of Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/ MLLM, Wright- Patterson Air Force Base, Prof. S.L. Semiatin, as well as with Prof. H.J. Rack. Sc. D., FASM, Department of ceramic & materials engineering.

There is interchange of scientific results and coordinated research plans.

What additional services can the ISTC provide that would assist you in reaching your

target?

- Assistance identifying Industry-attended Conferences
- Assistance clarifying a “graduation” or vision for “Industry Deployment” of your technology;
- Assistance identifying target Companies / Organizations.
- Assistance obtaining Business / Management Training;
- Assistance refining Market Intelligence;
- Databases access;
- Patent Support.

Private Sector Supplement

1. What are the possible applications of the technology (product) in the technological process / industry ?

What is the Industry Application Code of the application of your anticipated result (please consult the ISO's Industry Standardization Codes):

77.020, 77120, 77150, 11.060.15, 11.040.40., 47.040, 49.020, 49.025, 49.035.

What is the stage in the Technology Process or Cycle to which your anticipated Result relates:

1.ECA pressing to process billets Ø 20 mm with UFG structure out of VT6 alloy has been developed;
2.The prototype of novel deforming instrument for scaling ECAP billets Ø 40 mm has been designed and fabricated. The regimes of preliminary treatment of the initial structure for ECAP process have been established. Pilot billets with UFG structure have been processed.
3. Further investigations of effective preliminary treatment of structure in large-sized billets for ECAP, development of processing techniques for large-sized billets with UFG structure, optimization of construction, fabrication of effective ECAP die-set, processing of pilot billets, characterization of their structure and mechanical properties are viewed as very important tasks.

Please provide brief description of the known technological process (define) and its modifications

i Physical principles

Alternative methods of nanostructured (NS) materials processing are based on using methods of powder metallurgy, including nanopowders compacting.

ii Sequence of operations

Known methods of nanostructured (NS) materials processing include ultra dispersed powders compacting, obtained by various techniques: gas condensation in atmosphere of inert gas, plasmo-chemical method, aerosol synthesis, refinement in ball mill.

iii Schematic description of concrete technologies used in the designated technological process / cycle

Problems of known methods practical application are connected with small sizes of processing samples, preservation of some residual powder porosity at compacting, contamination of samples during powder preparation or their consolidation, as well as with difficulties of their application to the used metals and alloys.

Please describe the possible application(s) of technology (product) in the concrete technological cycle (define):

Unlike well-known methods of using severe plastic deformation (SPD), allowed to produce from VT6 bulk (for today up to Ø40x120 mm) nanostructured semi-finished products, suitable for practical application, for example, in medicine, aerospace industry, shipbuilding.

Specification	*ISTC Project 2125 p Result*	*Competitor 1* Semi-finished products with coarse-grained (CG) structure, processed by methods of traditional industrial technology.	*Competitor 2* A. Compound alloys B. UFG materials, processed by compacting nanopowders.
Accuracy			
Efficiency	*In medicine*: small sizes of implants with higher mechanical properties. *In aerospace industry*: enhanced operating characteristics of gas turbine engines, such as working time, take-off weight and others. *In shipbuilding*: decrease of construction weight.	*In medicine*: large sizes of implants with lower mechanical properties. *In aerospace industry*: lower operating characteristics of gas turbine engines. *In shipbuilding*: higher construction weight.	A.
Quality	UFG VT6: UTS = 1450 MPa, YS = 1420 MPa, Elongation = 11 %	Ti-6AL-4V. . UTS *1000* MPa, YS = 850 MPa, Elongation= 15 %, Ψ = 42 %.	B. Small sizes of processed samples, preservation of some residual powder porosity at compacting, contamination of samples during powder preparation or their consolidation.
Price			A. Higher price

Up to date titanium alloy Ti-6Al-4V is used as material to process details, used in aircraft construction (including propulsion engineering), shipbuilding, medicine and other branches of industry. Using Ti-6Al-4V alloy at high strength and plasticity will provide decrease of weight and enhancing of construction working time.

Please evaluate technical state of (technological process) in the industrially developed countries and RF. What is the status of technical development of (technological process) in the aforementioned countries (basic, applied research, research & development or industrial introduction.)

The investigation of severe deformation is a novel, but intensely developed scientific direction, the results of which have been discussed at recent conferences. But the given project on processing UFG alloy VT6 is one of two first works worldwide.

At the same time the researchers of ISSM (Ufa) performed the alternative investigations, financed also by EOARD. Method of multi-fold forging, used by ISSM, can also be viewed as a perspective one. There are some limitations in this method: higher labour intensiveness and some heterogeneity along the whole billet. Besides, the processed billet has the form of parallelepiped, that can be hardly subjected to further pressure treatment (rolling, pressing, drawing). On the contrary, in case of ECA pressing the billet has cylindric form, that is traditionally used for the subsequent treatment.

Which of the Companies / enterprises engaging in the (technological process) (ref. point ei above), provide analogous (competing) services products[1] at the same phase in the manufacturing cycle you have identified (point 1b of this supplement):
ISSM RAS (Ufa),
K.G. Farkhutdinov, scientific secretary, tel. (3472)-25-38-58.

List the Companies in your industry (point i. above), that provide <u>compatible</u> services products* <u>*near*</u>[2] the phase in the manufacturing cycle you have identified (point 1b of this supplement):

ISSM RAS (Ufa)

How would you characterize the Industry:

The technical result is competitive at the manufacturing market of aerospace, shipbuilding and rocket technics, implants for orthopedy, traumotology and dentistry.

Other application markets of UFG VT6 alloy will be investigated.

2. What is your “Graduation Strategy” and how is it justifiable?

What is the target market, the size of the market (in units and money), and what market share do you envision capturing with the industrial deployment of your technology?
Private companies for production of aerospace, rocket and shipbuilding technics, orthopedic and traumotologic centers and clinics of all industrially developed countries.

How would you characterize your resultant technologies:
- ❑ Revolutionary (no analogous technology on the market);

[1] or, investing in companies providing these service / products
[2] “near” means that is you have categorized your result as “component,” then what are companies in “sub-system integration,” for example.

- Incremental improvement / advancement in state-of-the-art: high strength and plasticity characteristics (e.g. increase in determining factors of competitive advantage – cost, sensitivity, operating conditions, other efficiency.)

How do you envision the commercialization / industry deployment of your anticipated results?

- Licensing

Result is licensing of Intellectual Property by the Owner(s), likely the Institute, which generates revenue for the Owner(s).

Which Companies from the above lists would be the most likely candidates for the type of relationship (section c above) appropriate for your Project Team's commercialization of the Technological Result(s):

EOARD

The Technological Results will be partnering application for patent "Processing technique for large-sized UFG billets out of Ti-6Al-4V alloy" and the subsequent licensing, which generates revenue for the Owner(s).

Public Sector Supplement

a) What types of Public organizations maintain an interest in the Development of this Technology?

Republic and area health care and medical technics organizations of Bashkortostan and Udmurtia republics, as well as Perm, Chelyabinsk and Samara regions.

What kinds of Public-sector organizations exist that fund the type of research (technical area) in which you are engaged?

- Foreign governmental organization (the Unites States Department of Energy)

- Russian Federation Government within the frame of Education Department programs, Russian Academy of Science, Republic Bashkortostan Academy of Science.

Do you know of other programming / organizations that would be candidates to fund the future development of your research? If yes, which ones?

Some enterprises of RF Health Care Department with public property form is ready to act as a consumer of nanostructured titanium semi-finished products for implants and medical instrument production: SEE CITO (Moscow), VNIPIMI (Kazan), «Medilar» (Krasnoyarsk), «Deosta» (Moscow).

Do you know what the eligibility requirements, proposal procedures, and approval timeline / procedures are for this / these organization?

There are known financing inquiry forms in some organizations, likely in Industry, Science and Technologies Department.

Which Public organizations have you targeted to fund the Research and / or Development of this Technology at your Institute?

RF Industry, Science and Technologies Department, RF Health Care Department organizations.

What are the requests for proposal (RFPs) and proposal submission / approval timeline for this / these organization(s)?

From 2004 to 2005.

What concrete plans / commitments / intentions have been made / explored with what Organization(s) for the implementation of your technologies / capabilities developed as a result of your ISTC project?

Participation in WEC exhibition, Moscow, 6 - 9th February 2002.

Participation in III Moscow International Salon of Innovations and Investments, 4 - 7th February 2003, WEC exhibition, Moscow;

Seminar “Bulk nanostructured materials: results of fundamental investigations and commercialization perspectives”, 5th February, 2003, WEC exhibition, Moscow. The seminar was organized within the frames of Salon in cooperation with RF Industry, Science and Technologies Department and consulting company “F&D”.

Do you have everything you need to accomplish your objectives vis a vis your project team's post-project sustainability?

We are planning partnering investigations and developments.
We are oriented on practical application.

What additional services can the ISTC provide that would assist you in reaching your target? :

New project fulfillment, aimed at investigation of effective (from the industrial manufacture) methods of fabrication of large-sized billets out of VT6 alloy (Ti-6Al-4V) with UFG structure.