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## **APPLICATION OF CRYOGENIC TECHNOLOGIES IN DEFORMATION PROCESSING OF METALS**

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The publications in the field of cryogenic-technologies' applications in the processes of the thermal and deformation treatments of metals are reviewed. The most effective fields of the applications of cryogenic liquids and gases for the heat treatment of working tools and metals (titanium, aluminium, and copper alloys) are in the rolling production and heavy engineering in order to improve the product quality, equipment and tool durability, to reduce the impact on the environment and operating personnel. The effects of cryogenic treatment and cooling on the tool life, wear, cutting temperature, surface roughness, dimensional accuracy, and cutting force are considered. As a result, the use of cryogenic processing and cryogenic cooling in machining processes increases the tool life and improves surface roughness as well as reduces the temperature of the machined surface, energy consumption during operation, and, thus, reduces tool wear that contributes to an increase in productivity. The possibility of obtaining and changing the nanostructure of a metal through the cryogenic cooling is also considered. The topic may be of interest for researchers and scientists in the field of metallurgy, materials science, and nanotechnologies.

**Keywords:** cryogenic cooling, liquid nitrogen, tool life, surface roughness, cryodeformation, low-temperature processing, nanostructured materials.

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## **1. Introduction**

The use of a cutting fluid (coolant) leads to a whole range of environmental, technical and economic problems. The need to recirculate, remove and dispose of coolant increases the cost of processing. In addition, liquids used lead to the occurrence of worker's skin diseases (dermatitis). The so-called dry machining (without or with minimal use of coolant), which has recently become quite widespread in the world, reduces tool life to a much greater extent than machining with coolant, and often negatively affects the structure of the resulting surface. Cryogenic treatment can be considered as one of the possible alternatives to the use of coolant in the processing, primarily of hard-to-machine materials, such as titanium alloys [1]. Potential benefits of cryogenic refrigeration include:

- sustainable production (cleaner, safer and more environmentally friendly);
- increased material removal rate;
- increase in service life of the tool;
- improving the surface quality of the machined part (quality/integrity of machined part) [2].

Attempts to deform metals at cryogenic temperatures began in the first of the 20<sup>th</sup> century, which turned out to be possible due to the development by that time of liquefying gases process (up to helium, 1908). Then, these works were continued on several metals. Experimental data on the use of cryogenic method in diamond turning of corrosion-resistant steels, turning of austenitic steels and titanium alloys, cast irons, grinding of bearing and carbon (low and high carbon) steels indicate a significant increase in tool life compared to using conventional coolant. However, the use of consumables such as liquid nitrogen and carbon dioxide increases the cost of cryogenic treatment, and therefore in many cases its economic efficiency remains in question, although the environmental value is undeniable. In addition, the maximum interest in the properties of cryodeformed metals and alloys occurred at the time of the start of the development of spacecraft and containers for liquefied gases (primarily hydrogen). Stainless steels were considered as the most interesting material. Their properties at cryogenic temperatures (strength, plasticity, hydrogen permeability, *etc.*) and the possibility of strengthening these steels by cryodeformation, primarily by cryorolling, were studied. The results obtained were of great scientific and applied interest, but, nevertheless, the amount of work on the cryodeformation of metals gradually decreased for two main reasons [3]. First, with a decrease in deformation temperature, plasticity of metal, as a rule, decreases, which makes it difficult to carry out necessary operation, namely, low-temperature rolling, drawing, *etc.* Secondly, plasticity of

the object is obtained under such an impact also decreases, and the more so, the greater deformation degree. To a certain extent, it can be restored by heat treatment, but with the loss of most increase in mechanical characteristics acquired during cryodeformation. In addition, disadvantages of the cryogenic method include the high price of liquid nitrogen, which is not subject to repeated use, in contrast to conventional cutting fluids, which usually circulate in machines for a week. Therefore, it is very important to choose an appropriate cryogenic refrigeration strategy to minimize usage and maximize efficiency [4].

The following key features of metals deformation at cryogenic temperatures can be distinguished:

- mechanical properties are usually lower than at room temperature in the early and middle stages of deformation;
- no generation of point defects, leading to blocking of both thermally conditioned diffusion and dislocation creep [5];
- at early and medium degrees of deformation, along with dislocation slip, alternative deformation modes (twin and stripe, stacking faults) are activated, which then disappear with the transition to a fine-grained structure;
- the process of fine-grained structure formation is greatly slowed down compared to room temperatures [6].

Cryogenic treatment is not the final operation and, to reduce the stresses caused by hardening or thermo-mechanical processing and obtain the required mechanical properties, steel after cryogenic treatment is necessarily subjected to tempering. Although cryogenic treatment refers to volumetric hardening methods, it is also effectively used on case-hardened steels, core of which must have high strength at high viscosity, and surface must resist abrasion well. In most cases, products with high carbon content in steel are subjected to cryogenic treatment, in the structure of which, after hardening or thermomechanical treatment, a large amount of residual austenite is retained [7].

## **2. Media Used in Cryogenic Processing of Metals and Alloys**

The idea of pressing with a liquid was first put forward by James Robertson in 1893 [3], and Bridgman was the first who had implemented this type of deformation [4]. The term ‘hydroextrusion’ for pressing materials with a liquid first appeared in the work of Pugh [8], after which it soon became generally accepted. With this type of action, all-round uniform factor forces the presence of compression during plastic deformation plays the most important role. However, the specificity of the method also carried limitations: it was considered natural that hydrostatic conditions for hydroextrusion can only be provided by liquid (considering pressure level being realized) media, which limited the

range of these media to liquids or metal melts. So, all work on hydro-extrusion was carried out at room or elevated temperatures. The range of media used at that time at different temperatures and in different pressure ranges was quite wide: pyrophyllite and talc, silver chloride and hexagonal boron nitride, indium and graphite. Certain hopes were placed on solid hydrogen and helium, although their significant compressibility is a big hindrance [4].

To implement large plastic deformations at cryogenic temperatures, such liquefied gas media are used: nitrogen, helium, carbon dioxide, etc. As the more suitable of the two liquefied non-combustible gases for use in cryogenic processing (nitrogen and carbon dioxide), nitrogen has significant advantages, since its boiling point is minus 196 °C (for carbon dioxide the temperature is minus 44 °C). Nitrogen tends to discharge (while carbon dioxide forms dry ice), which rises because it is lighter than air (carbon dioxide is heavier). In addition, nitrogen is cheaper than carbon dioxide. The cost of 1 l of liquid nitrogen is about \$0.085. The mechanism of liquid nitrogen action on the metal structure is not fully understood. In the study of several materials subjected to cryogenic processing, *e.g.*, when turning low- and high-carbon, bearing steel, titanium and aluminium alloys, it turned out that the workpiece should not be subjected to low-temperature cooling. In this case, workpiece metal increases its hardness, strength, abrasion in relation to the cutting tool, and preliminary sharp cooling increases the cutting forces arising during processing. Liquid nitrogen should be used to cool a tool (made of high-speed steel or hard alloy), which cryogenic temperature makes more solid and durable. When it was tested for shock loading, it also turned out that the brittleness of the tool material changes insignificantly, and its destruction does not accelerate [9].

### **3. Cryogenic Severe Plastic Deformation of Metals**

Cryogenic deformation of metals has been actively studied since the middle of the 20<sup>th</sup> century at the National Science Centre ‘Kharkiv Institute of Physics and Technology’ of the N.A.S. of Ukraine. In a large cycle of works, see, *e.g.*, [2, 6, 10–12], researchers revealed that lowering the temperature to the cryogenic region makes it possible to form structures of a high degree of dispersion in metals and alloys with a uniform distribution of defects in their structure. It was found that the main factor causing the formation of such structures is a change in the hardening rate (an increase in the dislocation density), leading to a faster increase in the average dislocation density than their density within the cell boundaries. A series of works, *e.g.*, [2, 10–12], revealed the most important role of lowering deformation temperature factor to the region of cryogenic temperatures, which led to the formation in

metal structures of such fineness with a high uniformity of defects distribution, which are unrealizable in the case of deformation at higher temperatures. Cryogenic liquids and gases are used in many industrial processes for processing materials of various properties for their fine grinding, granulation, low-temperature heat treatment, storage of food and biological products, in road and underground construction for concrete cooling and soil icing. If the exhaust air contains solvent, it can be cleaned by cryogenic condensation and the solvent can be recovered. In mechanical engineering, low-temperature processing, or cold processing, was first proposed in 1937 by Gulyaev [13] and later began to be developed [14]. For many grades of carbon and alloy steels (Y8, Y10, Y12, 9XC, IIIX15, XBГ, 40X13, X12M, *etc.*), complete end of martensitic transformation occurs at sub-zero temperatures, therefore, when cooled to room temperature a certain amount of austenite is staying. Reusable high temperature quenching cannot further change their structure. This means that the highest possible hardness value for given steel is not reached. In addition, retained austenite can gradually transform into bainite over time. Because of this, changes in the dimensions of finished products are possible. Therefore, for critical parts of precision equipment, bearings, high-precision measuring instruments, optics, *etc.*, it is desirable to convert austenite most fully to martensite. This is achieved by cooling to the end temperature of martensitic transformation, which can be in the range from plus to  $-140\text{ }^{\circ}\text{C}$  for industrial grades of steel. Increasing the carbon content over 1%, incl. when carburizing products and alloying steel, it reduces temperature of MT (by  $25\text{--}45\text{ }^{\circ}\text{C}$  for each 1% of alloying elements). Cooling below the MT temperature does not cause further transformation of austenite into martensite [15]. But, if the austenite stabilization temperature is  $>20\text{ }^{\circ}\text{C}$ , then, cooling after conventional hardening should occur as quickly as possible, otherwise the preserved austenite becomes stable after 1 h of exposure and then almost does not turn into martensite. Big quantity of carbon and alloying elements causes higher quenching temperature. Also, metals processing by cold studies results of on the of are considered in monographs [3, 5] at the E.O. Paton Electric Welding Institute of the N.A.S. of Ukraine a large complex of studies in the field of performance of welded joints at low temperatures was carried out, as well as studies on the low-temperature properties of metals at the I. M. Frantsevych Institute for Problems of Materials of the N.A.S. of Ukraine and the Gas Institute of the N.A.S. of Ukraine [6].

Cryogenic cooling is also used in the production of nanostructured metals using various types of severe plastic deformation (SPD), including cryogenic rolling. Deep deformation of metals (up to  $80\text{--}90\%$ ) is combined with heat treatment at liquid nitrogen temperature [9]. At the Donetsk Institute for Physics and Engineering named after O.O. Galkin

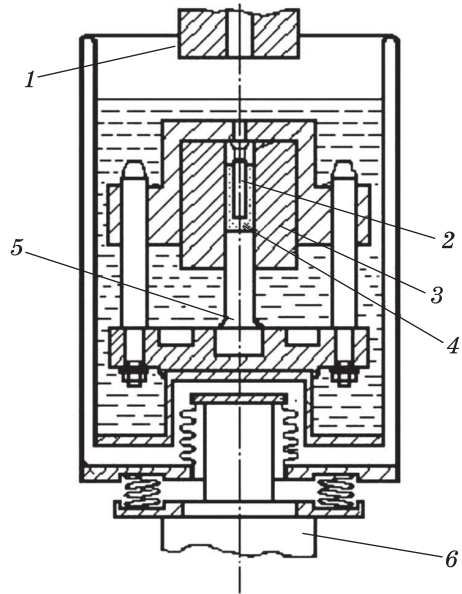
of the N.A.S. of Ukraine, such SPD methods as screw pressing and packet hydroextrusion are being developed [6]. Low-temperature deformation processes are studied at the B. Verkin Institute for Low Temperature Physics and Engineering of the N.A.S. of Ukraine [15]. Various types of low-temperature processing of alloys of rare and non-ferrous metals used in aerospace engineering, electronics and superconductors are considered. Such complex types of processing as vacuum [16] and cryogenic rolling [15], wire drawing in a cryogenic liquid with ultrasonic overlay [17] have been studied. To increase the metal resistance to cracking, a method of rolling in a hydrogen medium at the cryogenic temperature has been proposed at the G.V. Karpenko Physico-Mechanical Institute of the N.A.S. of Ukraine [18]. Industrial technologies for obtaining nanoscale structures for widely used steel grades using cryogenic liquid treatment are still under development.

The possibilities of using low-temperature quasi-hydroextrusion to improve the properties of structural materials have been tested to a large extent on stainless, austenitic steels in the initial state, first of all, on X18H10T. Such properties of this steel are known as significant ductility with strength characteristics sufficient for wide practical application: yield strength 220–240 MPa, tensile strength 620–650 MPa (at room temperature), transition of steel austenite during plastic deformation into a martensitic state (up to 20–25% at room temperature deformation, and up to 90% after stretching or compression at 77 K), and when heated with the fixation of interfacial boundaries. Stabilization of martensite leads to an increase in the strength of steel with some loss of plasticity [19].

Expected increase in strength characteristics due to a decrease in the temperature of hardening deformation was indeed present, but, as in the case of pure metals, differences were not just quantitative. As it turned out, the extrusion of steel at 77 K already by 30–35% led to the almost complete transition of austenite to martensite, and high dispersion. In the case of the quasi-hydroextrusion method using, the geometry of the forces acting on workpiece distribution is such that in the deformation zone the material is under the action of all-round compression forces. This plays a significant role, since the martensitic phase (both a and e) has a slightly less dense lattice than austenite (g-phase). The transition from the austenitic phase to the martensitic one under conditions of all-round compression is energetically unfavourable. But, if a combination of acting factors (a certain level of plastic deformation and a sufficiently low deformation temperature) still forces martensitic transition to occur, resulting martensite phase nuclei are deprived of the possibility of growth due to the action of all-round compression forces. It is the high density of interfaces in the practically single-phase structure of martensite that has to be explained by the fact that the processes of



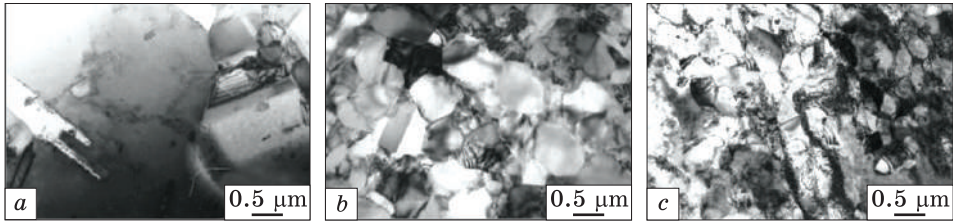
*Fig. 1.* Equipment for the low-temperature quasi-hydro extrusion (ICY-50 hydraulic press) [19], where 1 — stop-receiver, 2 — blank, 3 — high pressure container, 4 — pressure-transmitting medium, 5 — punch, and 6 — press table



boundary pinning associated with the precipitation of secondary phases (carbides and carbonitrides) occur in the case under study at temperatures that are 100–130 °C higher than those available in the case of annealing of the same steel deformed at the same (77 K) temperature, but with a different deformation geometry, in the absence of action of all-round compression forces in the deformation zone.

Although described methods provide steel with the ability to be elastically deformed up to 2000 MPa while maintaining a certain margin of plasticity, this is not of primary interest.

The f.c.c. metals were the objects of study at the initial stages of the low-temperature quasi-hydroextrusion formation as a powerful method of influencing metals by plastic deformation. But further development of the method required creation of new devices that would make it possible to search for opportunities to improve characteristics of structural materials that are widely used. This problem was solved by the development of several specialized devices. On the one hand, this is a low-temperature press with its own hydraulic power device, which, due to appropriate technical solutions, allows processing sufficiently large workpieces without significant coolant costs [11]. On the other hand, these are attachments to standard industrial presses that allow you to work both with refrigerants that require a closed circuit for evaporating gas, and with an open Dewar vessel. Figure 1 shows a diagram of the latter, used as a low-temperature extrusion attachment to the PSU-50 press. Possibilities of using low-temperature quasi-hydroextrusion to improve the properties of structural materials have been tested to a large extent on stainless, austenitic steels in the initial state, first of all, on X18H10T. Such properties of this steel are known as significant ductility with strength characteristics sufficient for wide practical application: 220–240 MPa yield strength, 620–650 MPa tensile strength (at room temperature), transition of steel austenite during plastic deformation to a martensitic state (up to 20–25% at room tem-



*Fig. 2.* The images of Cu microstructure in the initial state (*a*), after the ECAP via standard technology (*b*), and after the ECAP with cryogenic nitrogen cooling (*c*) [21]

perature deformation and up to 90% after stretching or compression at 77 K), and, when heated, fixation of interfacial boundaries, stabilization of martensite lead to an increase in the strength of steel with some loss of plasticity.

Variation of methods and technological modes of thermomechanical treatment, cryogenic treatment and subsequent tempering provides a different structural state and a combination of mechanical properties of the metal. For example, during the hydroextrusion of pure copper, there is a transition from the classical block (or grain) structure to a lamellar structure with a plate thickness of several fractions of a micron [20].

As assumed in Ref. [21], lowering the temperature of the equal channel angular pressing (ECAP) should suppress the processes of dynamic retrogression and recrystallization, preserve a high dislocation density and activate mechanical twinning promoting additional plastic deformation of copper. This should raise the efficiency of the deformation treatment and hence affect structure and physical and mechanical properties of copper. The microstructure of copper in the initial condition is imagined in Fig. 2, *a*. The mean grain size amounts to 53  $\mu\text{m}$  with allowance for presence of twins. Metallographic analysis of metal after six passes of ECAP with cooling at room temperature and in liquid nitrogen has shown in Fig. 2, *b*, *c* that microstructure does not undergo principal changes upon lowering of deformation temperature from room to cryogenic one. The first pass produces an elongated structure with strongly smeared curved boundaries. With growth in the number of passes, the boundaries become better manifested, but preserve considerable thickness; grains remain chiefly elongated. After the fourth path, the structure starts to be equiaxed, but many boundaries remain smeared. After six cycles, the structure is quite homogeneous, equiaxed and contains strongly deformed subgrains in both cases. With the use of nitrogen, structure becomes more fine-grained, because the cryogenic treatment suppresses dynamic retrogression due to the lower mobility of dislocations and increase of their density. Therefore, the structure formed by the ECAP with cooling in nitrogen is characterized by diffuse, nonequilibrium and poorly manifested grain boundaries that is



shown in Fig. 2, *c*. Distinctive feature of the structure formed by ECAP with cooling at cryogenic temperature is the formation of dislocation clusters. After the ECAP at room temperature, the dislocation density is lower and grain boundaries are defined better [21].

An analysis of the literature data showed that the cryogenic deformation of pure metals is the most studied, while the regularities of cryogenic deformation of two- or more-component mixtures of pure metals, as well as the effect of changing the temperature regime on the solubility of the components (depending on the enthalpy of mixing, characteristics of structure and their properties) are insufficiently studied and are of scientific and practical interest.

### **3.1 Machining of Titanium Alloys**

Metal cutting, such as turning, milling, *etc.*, is a form of subtractive manufacturing where a sharp tool is used to remove physically material to achieve the desired geometry. During machining, heat is generated, which limits the life of the cutting tool and affects part quality and cutting forces. Many researchers have studied the mechanism of heat generation in metal cutting to optimize machining process with good part quality and long tool life [22]. Titanium alloys have always attracted interest due to their wide range of applications in the aerospace, automotive, chemical and medical industries. Cryogenic cooling, which is an environmentally friendly alternative to traditional emulsion cooling, is an effective way to maintain the temperature at the cutting edge well below the softening temperature of the cutting tool material [22].

It is now well known that in order to achieve a finer grain size, the lowest possible deformation temperature [21] should be used. To date, all successful ECAP processing of titanium has been performed at or above room temperature, with the exception of one paper that reported advantageous processing of titanium in three passes at 77 K using a die with a channel angle of 90° [23]. Figure 3 [25] shows a matrix with a channel junction angle of 120° and pressing pure titanium at cryogenic temperature in four passes. Titanium specimens 9 mm in diameter and 72 mm in length were cut from annealed material and inserted into 6061 Al alloy pipes with a wall thickness of 3 mm by interference fit. Before each pass, samples were immersed in liquid nitrogen for 10 min. Also, to minimize the effect of thermal diffusion between samples and the matrix walls, the matrix channels were cooled with liquid nitrogen during each pass [25].

The effect of different approaches in cryogenic cooling in Ti–6Al–4V was studied in Ref. [26]. As shown in Fig. 4, different cooling approaches result in different cutting tool temperatures (measured and predicted). Cooling in descending order of efficiency (from worst to best) is: dry cutting, cryogenic tool re-cooling, emulsion cooling, pre-

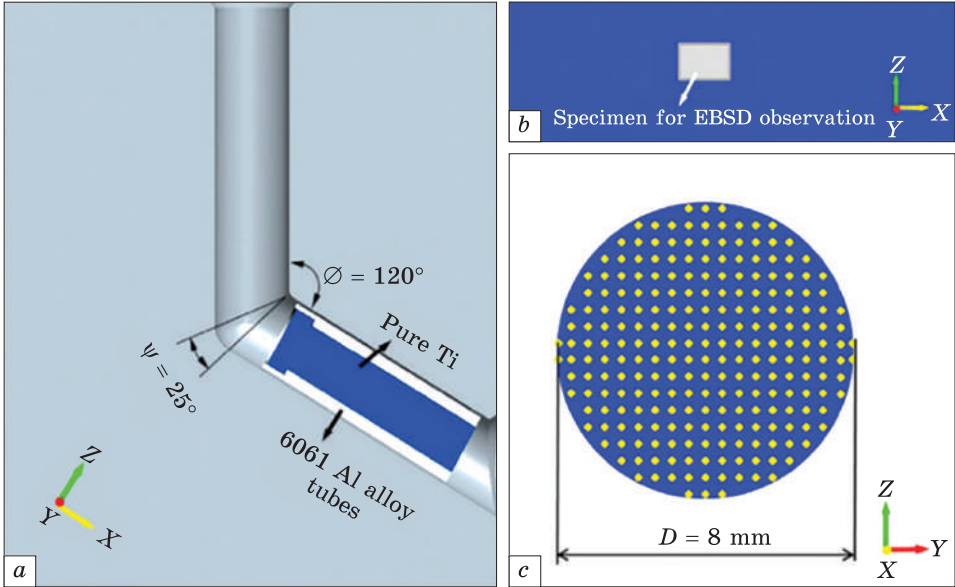


Fig. 3. The equal channel angular pressing process sketch (a), orientation of the sample to observe the electron backscattered diffraction (b), and pattern for the Vickers microhardness measurements on the cross-section (c) [25]

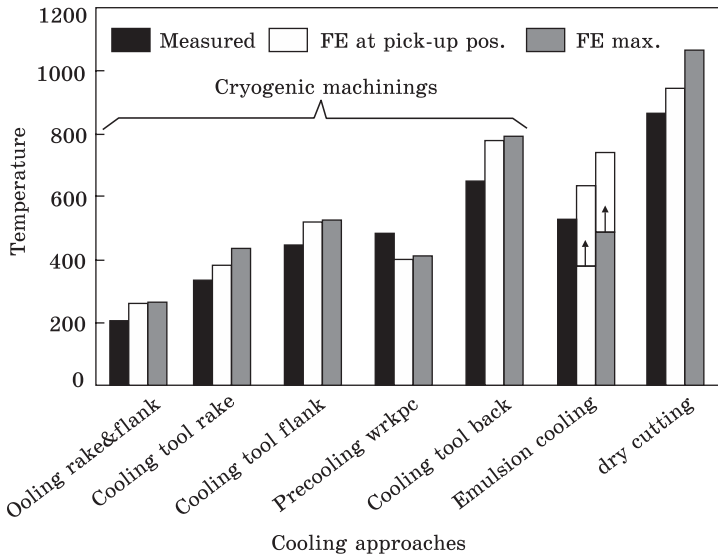


Fig. 4. Measured and predicted tool temperature for different cooling methods [26]

cooling of the workpiece, cryogenic side cooling, cryogenic doctor blade cooling and simultaneous doctor blade and flank cooling [22]. The study also explored modified tools to increase the efficiency of cryogenic cool-

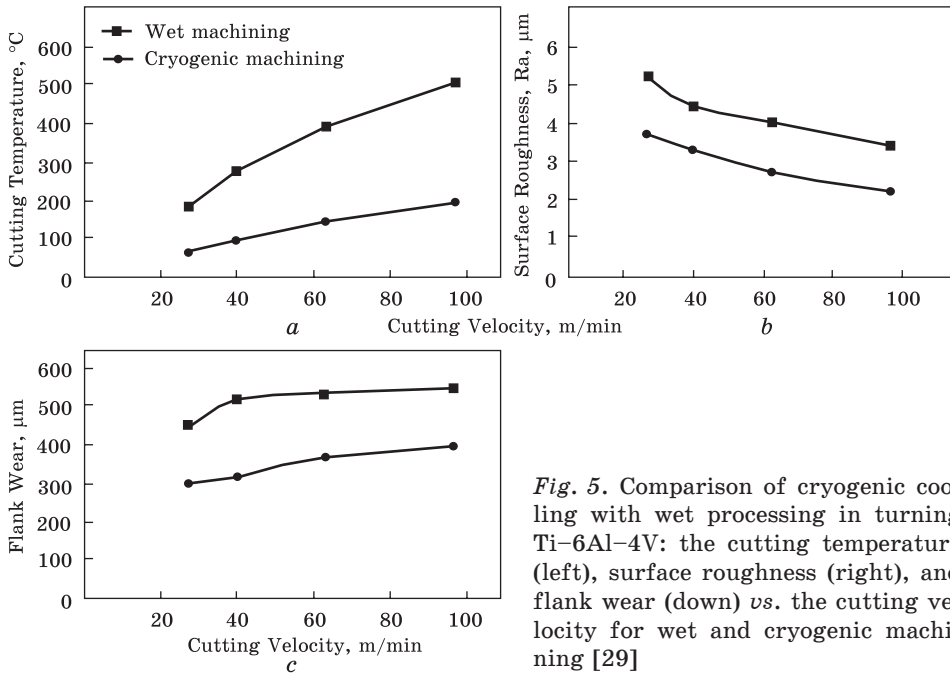


Fig. 5. Comparison of cryogenic cooling with wet processing in turning Ti-6Al-4V: the cutting temperature (left), surface roughness (right), and flank wear (down) vs. the cutting velocity for wet and cryogenic machining [29]

ing in order to maximize efficiency. Instead of flooding the overall cutting area, a liquid nitrogen supply system is used, spraying nitrogen only on a localized area of the tool nose and/or tool side in well-controlled jets. All cryogenic machining approaches result in a higher basic cutting force in comparison with dry cutting. The more nitrogen used, the lower the cutting temperature. Cryogenic cooling tends to increase the cutting force because work material becomes harder and stronger at lower temperature, while the lower temperature makes material less sticky, decreasing the cutting process' internal frictional force [22, 27]. The data obtained are agreed with measured and calculated temperatures as it is shown in Refs. [22, 27].

Other authors [28, 29] also used modified Ti-6Al-4V turning inserts and compared the cryogenic cooling approach with wet machining from different angles. For wet processing, the coolant emulsion was obtained by mixing the concentrate with water in a soluble oil ratio of 1:20. The results regarding cutting temperature, surface roughness and flank wear for wet and cryogenic machining are shown in Fig. 5. The tests were carried out at various cutting speeds while maintaining a constant depth of cut and feed rate (1 mm depth of cut, 0.159 mm/rev feed speed, 27, 40, 63, and 97 m/min cutting speed) in Fig. 5 (left). It was observed that during cryogenic cooling, cutting temperature was reduced by 62% compared to wet machining due to the direct supply of liquid nitrogen to the heat generation zones through the holes made in

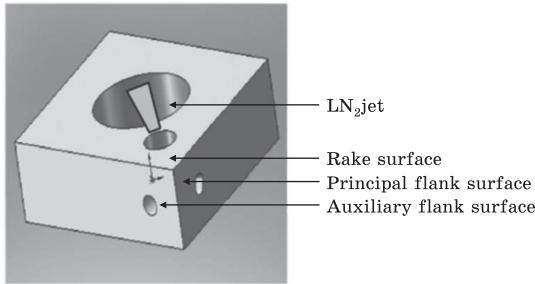


Fig. 6. Modified cutting tool: the sketch illustration [29]

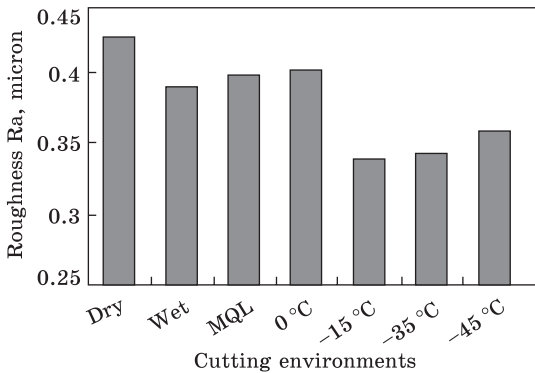


Fig. 7. Effect of different cutting media on the roughness [7]

cooling using a minimum amount of liquid nitrogen injected through a micronozzle formed between the chip breaker and the front cutter, using a secondary back face-cooling nozzle, presented on the Fig. 6 [22, 26]. Thus, liquid nitrogen absorbs heat and evaporates, forming a liquid-gas cushion between the chips and the tool surface, which acts as a lubricant. The lubricating effect and effective cooling help to reduce cratering and flank wear. In addition, liquid nitrogen is not wasted on unnecessary cooling places and eliminates the negative impact of increased cutting forces and abrasion during precooling of the workpiece material. Since nitrogen consumed is less, this concept is called economical cryogenic treatment.

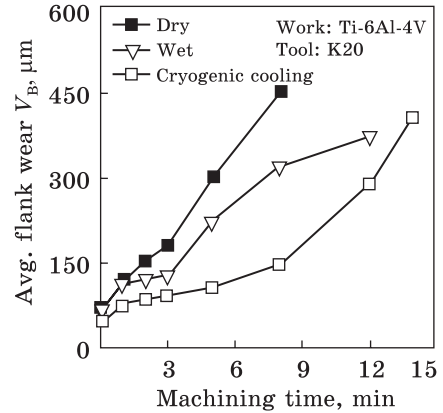
As mentioned earlier, cryogenic refrigeration can be applied to other liquids such as an air. The effect of cooling air temperature on cryogenic treatment of Ti-6Al-4V in combination with a minimum amount of lubricant has been studied in Ref. [7]. They experimentally studied cutting force, tool wear, surface roughness and chip morphology to compare the effects of different cooling air temperatures. As seen in Fig. 7, cooling at temperatures below 0 °C significantly reduces flank

the cutting insert [28]. Figure 5 (right) shows the average surface roughness Ra obtained after wet and cryogenic treatment. The reduction in Ra due to cryogenic cooling was about 25–35% compared to wet processing due to less adhesion between newly created surface of the workpiece and auxiliary side surface of tool and a lower tool wear rate, which is also seen in Fig. 5 (down). As concluded [29], the cryogenic cooling showed a significant improvement in cutting force, surface roughness and tool wear by controlling the temperature of the cutting zone with a carefully modified tool for maximum efficiency.

As the economical cryogenic refrigeration concept showed, the tool life was increased 5 times by emulsion

Fig. 8. The average surface wears vs. the processing time [30]

wear, which is also visible on the scanning electron microscopy (SEM). Authors of Ref. [7] also report that in terms of surface roughness, minimum quantity lubrication (MQL) with  $-15$  and  $-30$  °C results in better surface quality, probably due to better lubrication and cooling effects, resulting in lower tool chip friction. The roughest surfaces are obtained with dry cutting due to more intense temperature and friction between the side surface of the tool and the workpiece [7, 22].



Authors of Ref. [30] compared tool wear with an uncoated microcrystalline WC/Co insert for dry and wet cryogenic turning conditions (Fig. 8). Liquid nitrogen was used for cryogenic cooling. Tool wear is measured by scanning the entire area of the crater. The authors concluded [30] that the crater and flank wear of WC/Co inserts is the result mainly of dissolution-diffusion, and in the case of other tool materials, abrasion is the main cause.

To understand the effect of cryogenic cooling on friction, an idealized disc-to-plane test contact for various liquid nitrogen applications has been performed, as illustrated in Fig. 9 [27]. Initially, it was assumed that the liquid nitrogen lubrication mechanism was due to a decrease in friction and due to a change in material properties upon cooling. However, tests have shown that this is not always the case, and the effect is highly dependent on the material pairs. The second assumption prior to testing was that the injection of reduced nitrogen into the contact zone creates a film, and testing confirmed that the liquid nitrogen jet was very effective in reducing friction. Another important test result was that the coating layer as a solid lubricant gives good results in

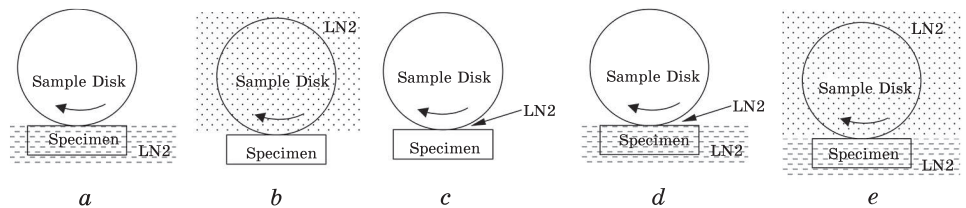
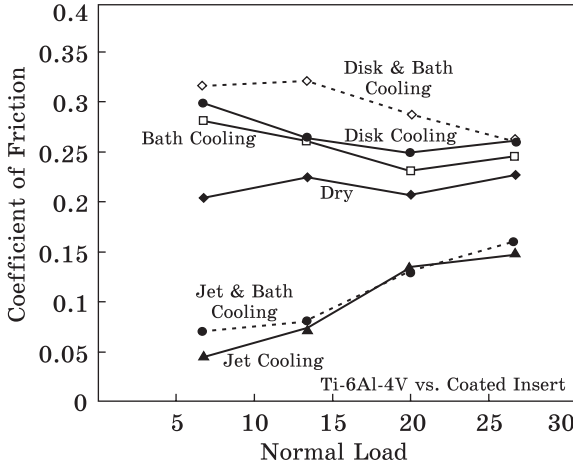


Fig. 9. Different cases (a–e) of using liquid nitrogen between two materials: a – cooling in a bath, b – disk cooling, c – jet cooling, d – jet and bath cooling, e – disk and bath cooling [31]



*Fig. 10. Friction coefficient for Ti-6Al-4V with different cryogenic treatments [31]*

machining, but this can lead to adverse lubrication effects at low temperatures and when cooling with liquid nitrogen of uncoated plates. Figure 10 [27] summarizes the results obtained with Ti-6Al-4V against a coated insert at a speed of 0.3 m/s.

Based on the above results presented, it can be summarized that friction factors determine abrasion and cutting forces in cryogenic machining. On the one hand, if liquid nitrogen is applied properly, the lubricating effect can reduce the friction. On one side, higher cutting speeds or thermal softening reduces the friction between tool and workpiece, and cryogenic cooling hardens the workpiece and minimizes thermal softening that enhances friction. The overall conclusion is that selecting the right combination of feed rate and depth of cut has the greatest impact on tool life.

### **3.2. Cryogenic Deformation of Aluminium and Al-Based Alloys**

Some researchers argue that in the process of cold (and cryogenic) deformation of aluminium and its alloys with large degrees, a grain structure similar to the structure formed in aluminium and its alloys subjected to SPD can also be formed. Thus, during cryogenic all-round forging of the 7075 alloy (Al-5.6Zn-2.5Mg-1.6Cu, wt.%) [31], the formation of the ultrafine-grained/nanocrystalline (UFG/NC) structure with an average grain size of  $\approx 500$  nm was observed. As stated in Ref. [32], cryocooling of the same alloy with a degree of deformation of  $\approx 80\%$  led to the formation of an NC structure with an average crystallite size of  $\approx 100$  nm. However, it should be noted that the conclusion about the formation of precisely grains with high-angle boundaries in these works was made only based on data from a qualitative analysis of transmission electron microscopy (TEM) images, which casts doubt on the very fact of their existence, since such an analysis fails to quantitatively separate crystallites into grains and subgrains, according to their mutual misorientation and volume fraction.

It is usually believed that the process of formation of new grains during cold and cryogenic deformation proceeds largely by mechanism

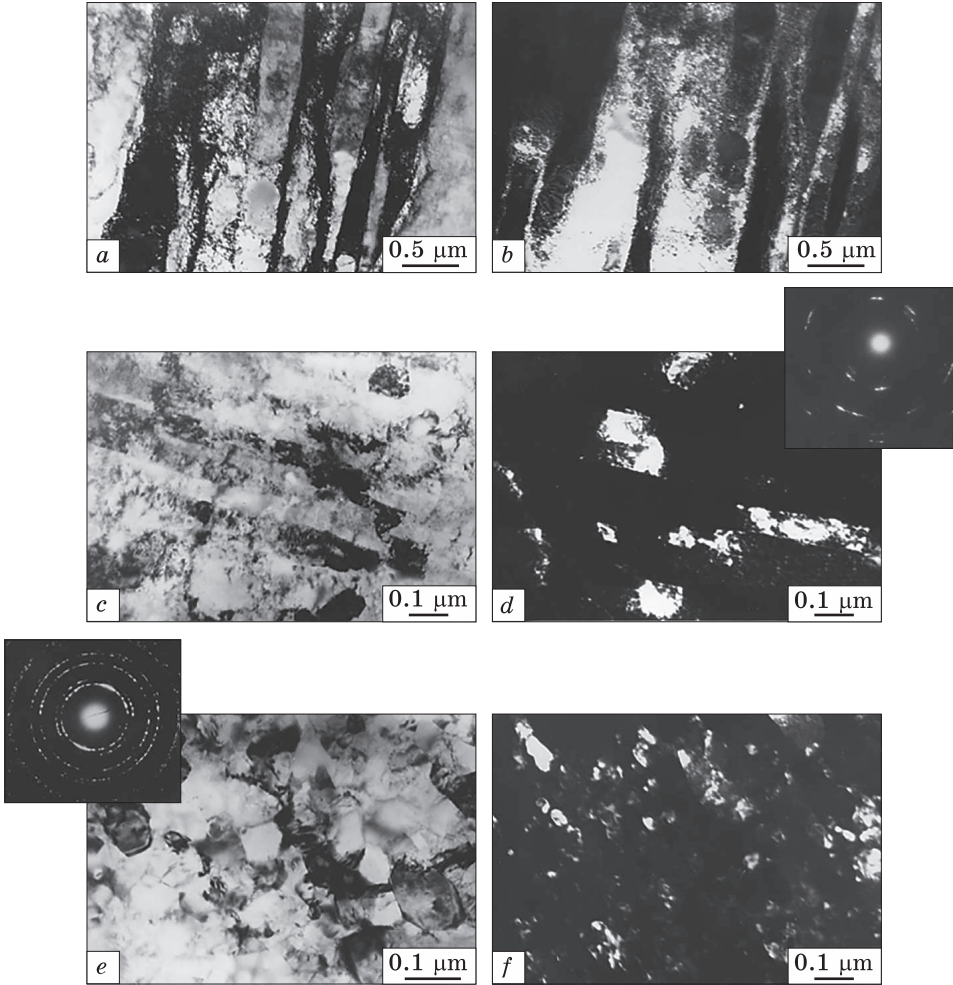


of continuous dynamic recrystallization (DR). The latter differs from the static one in that the recrystallized grains with a low dislocation density that have appeared are constantly riveted due to ongoing deformation. DR under cryogenic temperatures can begin at lower degrees of deformation than at room temperature. Thus, in Ref. [33], DR during cryorolling of Al 6063 alloy (Al–0.45Si–0.3Mg–0.015Cu–0.013Mn–0.058Fe–0.022Zn–0.02%Cr, wt.%) was already observed at a degree of deformation  $e \approx 3.8$ , while, *e.g.*, in the Al–Mg–Li alloy during ECAP, it was fixed after the total degree of deformation  $e \approx 10$  [34]. It is known that the critical degree of deformation required for the onset of DR increases with an increase in the Zener–Hollomon parameter, which is inversely proportional to temperature. In this case, it will also proceed at a lower temperature if the stored strain energy reaches a critical value. Since a significant part of the deformation energy at room temperature is converted into heat during adiabatic heating of the sample, during cryogenic deformation, a much larger part of deformation energy is stored in the form of defects in the crystal structure, which, in turn, can reduce the critical degree of deformation [35].

Several works on cryodeformation [36–38] show the effect of deformation temperature on other types of defects. During cryogenic hot isostatic pressure (HPT) treatment of b.c.c. Fe, the mobility of dislocations becomes low, because of which, along with dislocation glide, twinning and the formation of deformation microbands become acting mechanisms of deformation [35]. As a result of changing the mechanisms of plastic deformation, there is no unambiguous correspondence between structure and calculated degree of deformation, deformation process becomes inhomogeneous and uneven. Thus, during low-temperature deformation of cobalt (h.c.p. lattice), stacking faults, fault bands, and multiple twinning are formed at intermediate stages, which disappear with increasing deformation, giving way to a homogeneous submicrocrystalline structure, both with high-angle and low-angle misorientations [37]. For Ni [37], dislocation slip also slows down and twin and microstrip modes are switched in Fig. 11.

As the degree of deformation increases, the twins and microbands are fragmented and then disappear. Hardness in the early and middle stages of deformation is lower than at room temperature due to the inhomogeneously formed deformation structure, both with a submicrocrystalline grain size and with larger grain sizes, and with their different mutual misorientations. In Cu, up to 10 anvil revolutions, with the preservation of unfragmented twins and dislocation cells, no uniformly distributed submicrocrystalline structure was detected [39].

Cryogenic treatment in metal forming processes in the production of forging dies and other tools, improves the formability, strength and surface quality of the sheet [15], the cryogenic process can effectively



*Fig. 11.* Effect of the hot isostatic pressure cryodeformation on the Ni microstructure for different values of the strain degree: 0.5 (*a*, *b*), 5.3 (*c*, *d*), 5.5 (*e*), and 6.7 (*f*) [38]

improve the tensile strength and yield strength of aluminium alloy by greatly increasing dislocation hardening and hardening of grain boundaries. The microstructure, mechanical properties, annealing behaviour, and post-annealing microstructure/performance relationship of 5052 Al alloy piece material was studied by cryogenic rolling [40]. They obtained information after analysis that, compared with rolling at room temperature, cryogenic treatment can effectively improve the tensile and yield strength of the 5052 Al alloy. Cryogenic process for the 5052 Al alloy was  $\approx 30$  MPa higher than that for the rolling process at room temperature. In the work [41], the Al 6061 melt was cryorolled to a thick-

ness of 85%. The Al 6061 alloy solution was treated at a temperature of 530 °C for 3 h, followed by water quenching to room temperature. Next, samples were immersed in liquid nitrogen 20 min before the first rolling pass and 5 min before each rolling pass in the cryorolling process. They obtained result that when applying cryorolling process on Al 6061 alloy, dislocation density increases due to the suppression of dynamic rolling recovery. As a result, tensile strength and hardness of the sample will increase. Authors of Ref. [42] investigated AW-6016-T4 cryogenic sheet metal in the temperature range from -196 to 25 °C at uniaxial stretching with Nakazima tests to study the formative behaviour. Samples were cooled by spraying liquid nitrogen into the chamber until the sample reached the desired temperature. Cryogenic forming has shown improvements in both strength and part quality. Previously, maximum depth of 6 mm was achieved with the room temperature moulding, while a maximum depth of 8 mm was achieved during cryogenic moulding at -150 °C. This research demonstrates potential of cryogenic moulding for the production of complex automotive components. The effects of cryogenic treatment on AISI D2 steel stamping equipment were studied in Ref. [43]. As a result, about 3.5% of the annual turnover cost was saved and production was increased by 60%. Technologies for the use of liquid nitrogen for cryogenic processing of workpieces are environmentally friendly and safe.

In order to study the factors that limit the production of real nanograin materials by cryogenic severe deformation, grain structure formed in Al-0.1%Mg alloy under in-plane compression at temperatures up to 77 K and preliminary SPD by equal-channel angular extrusion was studied. Change in the deformation mode in itself had little effect on the increase in the rate of grain refinement. At the minimum cryogenic temperature (77 K), samples still contained 30% low-angle boundaries, and the distance between high-angle grain boundaries (HAGB) was obtained in only one dimension. At large deformations, steady-state minimum distance between HAGBs approached regardless of the temperature at which the grain refinement rate remained unchanged. It is shown that minimum grain size achievable in SPD is limited by the balance between the HAGB distance compression rate and dynamic grain coarsening. At low temperatures, this is controlled by anomalously high rates of boundary migration, which are difficult to explain by existing theories of grain boundary mobility [40].

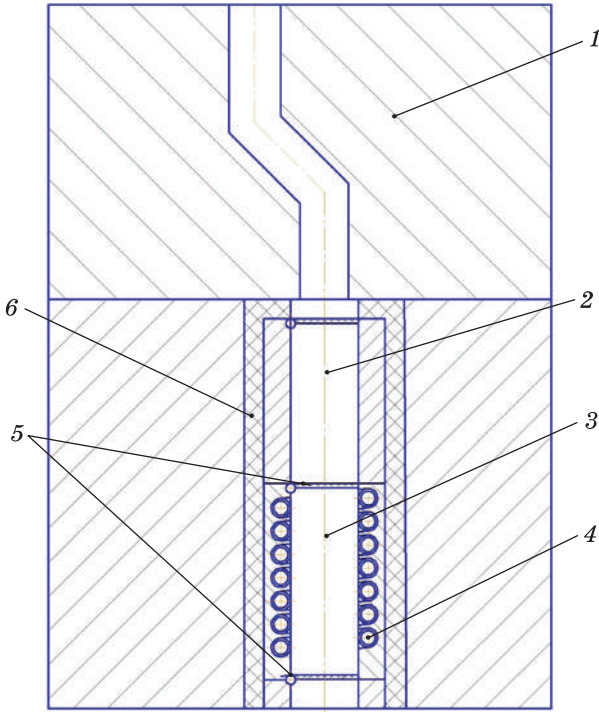
Nowadays, SDP has been used successfully to produce submicron grain patterns, and in some cases nanoscale crystallites, in a large range of alloys (*e.g.*, [36, 37]). However, an important restriction of SDP is that the grain-milling rate becomes increasingly inefficient at higher stress levels [40]. Even in conventional high strain rolling, the high angle boundary distance reduction rate decreases to a very low rate at

quench stage IV [24], and at higher strains achieved with SPD techniques approaches a grain structure at steady state [37, 38]. For example, during equal channel angle extrusion (ECAE) of aluminium alloys, grain refinement reaches a limit when the HAGB distance converges with the subgrain size, and the area fraction is typically 70–80% saturated [7, 19]. When deformed by the ECAE method at room temperature, authors of Ref. [44] reported that a small decrease in the transverse distance remained. However, a true steady state was not reached and there was still a marked reduction in grain length due to breakage of the final remaining length as well as fibrous, ribbon-like grain fragments. However, in general, only small changes in grain size were seen compared to SPD when the applied strain is increased by a factor of 10 [1, 35]. Stationary grain sizes were also subjected to ultra-high deformation, like a cryogenic ball mill. Thus, there is a minimum grain size achievable in a material with a given SPD process under constant deformation conditions [1]. Since grain-grinding speed becomes more inefficient with stress, changing the warp mode can help spice up the generation of new living space. For example, changing the deformation mode can change the development of texture and increase the compression ratio of centre distance exponentially by required applied deformation. It has been documented that a stationary subgrain size is observed during warm deformation of metals due to dynamic recovery. In highly deformed alloys, constant grain size at ultrahigh deformations is explained by dynamic recovery. However, as noted in Ref. [45], recovery processes are currently poorly understood, especially at low temperatures, when the boundary migration rates required to maintain a constant grain size are orders of magnitude higher than can be satisfactorily explained by diffusion control. Alternative mechanisms have been proposed for the rapid deformational growth of grains, including stress-induced boundary migration and detachment from solute anchorage. As in the case of subgrain [26], the minimal grain size attainable during SPD is generally associated with the temperature compensated deformation rate during processing and is specified by the Zener–Hollomon parameter [44]. For this reason, it is achievable to obtain a reduced grain size by the deformation temperature decreasing, which causes suppression of thermally activated reductive processes. Cryogenic deformation using led to the nanocrystal structures formation in Al, Cu and other alloys, which offers practical perspectives for the preparation of bulk nanogranular materials by the SPD method. As a rule, the materials involved are high rolling materials at liquid nitrogen temperature that have already been strained by the severe deformation method, such as ECAE [34]. It is also claimed that nanograin structures can be prepared by direct cryogenic rolling. However, most works have considered copper samples, where twinning is an additional source of grain fracture. As a result of the

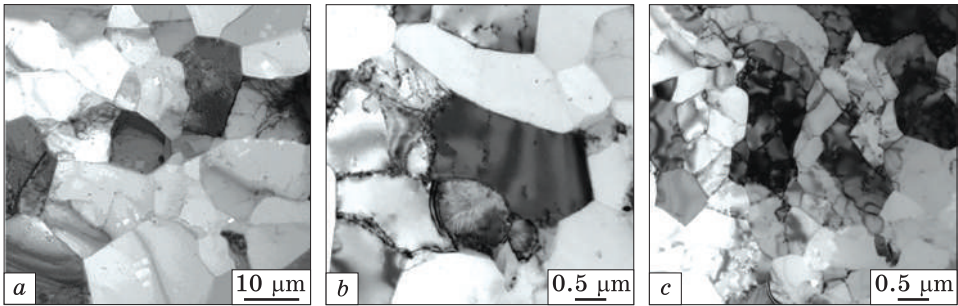
small size of crystallites formed by cryogenic rolling of SPD, most of the information reported in the literature is based on the TEM data. As a consequence, it is often not possible to obtain reliable statistical data with sufficient resolution to characterize fully the disorientation of boundaries in materials throughout a representative grain assembly. Thus, SPD method contributes to the creation of nanograined structures, since they have a HAGB distance in all dimensions of less than 100 nm, or still contain a large proportion of low-angle boundaries (LABs). In addition, deformation of a structure obtained by compression by planar deformation is usually highly elongated [1, 32] and is unlikely to have nanoscale HAGB distances in all directions. The main goal of the study presented here was to investigate the effect of changing the deformation mode and lowering the temperature on the grain refining rate and grain size limit achievable with the SPD process. This was achieved by studying the evolution of the deformation structure in a dilute aluminium alloy that is cryogenically deformed under plane deformation conditions over a range of temperatures, after ECAP pretreatment [35].

In Ref. [24], the combination SPD at room temperature with the ECAP method with liquid nitrogen cooling of workpiece immediately after leaving the die. Theoretical basis of this processing method refers to the representation of all crystalline bodies' plastic deformation process as a physicochemical transformation accompanied by recrystallization in the process of deformation. Aluminium alloy 2024 was the material studied because it was easy to tune parameters of secondary phase, including their modality, morphology and distribution size, to change the structure of stamp from coarse-grained to recrystallized, including UFG sample. Experimental sample belonged to a hot-pressed rod with a diameter of 30 mm from an industrial deformable heat-strengthened alloy 2024 of a standard chemical composition (Al-4.4Cu-1.4Mg-0.7Mn, wt.%). Workpieces of square section from  $15 \times 15 \times 70$  mm<sup>3</sup> cut along the axis of rod were heated to 500 °C and after half an hour quenching in water to fix supersaturated aluminium solid solution. Resulting samples were further subjected to ECAP in a conventional array of parallel channels with a 125°-intersection angle. ECAP was carried out along the route with the workpiece rotated 90° around the longitudinal axis. Friction between the tool and workpiece was reduced by using palm oil with graphite as a lubricant. Deformation was carried out at room temperature. In the first series of experiments, each blank was deformed by the ECAP method. In the second series of experiments, after each conditional passage of workpiece through the die, it entered a container with liquid nitrogen presented on the Fig. 12. In both cases, the number of passes through the array of parallel channels was 4. To analyse the structure of the prepared sample, the TEM was used. Microstructure of





*Fig. 12. The ECAP sketch with liquid nitrogen cooling. Here, 1 — die with 3 channels of the same cross section 2 of which (the inlet and the outlet ones) are parallel to each other and the middle channel is inclined with respect to the inlet and outlet channels, 2 — intermediate chamber, 3 — quenching chamber, 4 — system for nitrogen circulation, 5 — shutoff components, and 6 — thermal insulation [24]*



*Fig. 13. Microstructure of Al 2024 in the initial state (a), after four passes of ECAP (b), and after four passes of ECAP with liquid nitrogen cooling (c) [24]*

the alloy in the initial state, after quenching at a temperature of 500 °C with cooling in water is shown in Fig. 13, *a*. Structure is a supersaturated solid solution based on aluminium and undissolved phases of crystallization and eutectic origin. It is necessary to compare microstructure of alloy before and after deformation to evaluate the effectiveness of ECAP and effects of cryogenic cooling. Photographs of microstructure after four pressing cycles under various cooling conditions are shown in Fig. 13, *b, c* [24]. Metallographic analysis of alloy after ECAP in a die of parallel channels structure at room temperature and during



cooling in liquid nitrogen shows that strongly deformed grains/subgrains are formed in both cases. However, in experiments with nitrogen, structure is more dispersed with a smaller grain size, evident in Fig. 13, *c*, since cryogenic treatment suppresses temperature reduction process characteristic of aluminium in the room. Movement of dislocations is limited during this treatment and therefore dynamic recovery is inhibited. Therefore, the microstructure obtained after ECAP using nitrogen is characterized by diffusive processes, nonequilibrium and weakly expressed grain boundaries, which only ultimately should lead to the equilibrium state because of the relaxation kinetics [46–51].

#### **4. Heat Treatment of Working Tools**

The costs of used cutting fluid (coolant) recycling can exceed its initial cost by half. Therefore, leading manufacturers of machine tools began to master the processes of dry machining using cryogenic liquids as a coolant [26, 27]. The most effective supply of cryogenic coolant is directly to the top of tool. At the same time, workpiece will retain a generally constant temperature, which will prevent its dimensional deviations and geometric deformations, as well as an increase in its hardness and strength in relation to the cutting tool. Liquid nitrogen is supplied through a micronozzle located between the front surface of the cutting insert and chip breaker, which should raise the chips so that they do not interfere with the coolant jet entering the contact zone, where the maximum amount of heat is generated. There it quickly evaporates, forming a ‘cushion’ of gas and liquid on the surface of tool and causing a lubricating effect. Liquid nitrogen is supplied from a standard heat-insulated cylinder at a pressure of 14–23 bars. Flow rate of coolant is such that one bottle of standard capacity is enough for a whole day of machine operation [52]. The analysis showed that with an increase in the cutting speed, the drop in tool life with the use of liquid nitrogen increases several times compared to the coolant, i.e. it is most effective in high-speed processing. In addition, when machining on automatic machines, chip formation and welding play an important role in ensuring process stability and improving heat dissipation. In Ref. [53], kinematic characteristics of turning an austenitic class material process were studied. A method and device for local cryogenic impact (LCI) has been developed, dynamic model for assessing the stability of chip segmentation and an algorithm for automating the choice of LCI parameters have been proposed. In general, considering all the operational and cost factors of cryogenic cooling, its use is more profitable than standard coolants.

When stamping products of complex shape, along with local heating of the tool, its local cooling is used to increase the strength of the deformed material in dangerous sections. This allows you to significantly

increase the degree of deformation in one transition and, consequently, increase productivity. Cooling time is up to 20 s; limiting drawing ratios for steel 10 and 20 in this process reach 3.0 [53].

Cryogenic liquids and gases are also used as the most efficient coolant with high specific heat release in the processes of plasma spraying of coatings, which allows not only to intensify, but also to improve the quality of surface treatment [54].

SPD methods can effectively grind microstructure of most structural materials only to a certain small grain size (usually up to the SMC interval), after which the grinding process gradually fades. The reasons for this phenomenon are not entirely clear. Sometimes this is associated with the onset of a certain balance between the deformation reduction of grains and their thermally activated growth. Thus, it is not yet possible to achieve guaranteed formation of real NC structures by SPD in most cases, and it is necessary to look for new ways to achieve the NC grain size range. One way to solve this problem can be deformation at very low temperatures — the so-called cryogenic deformation. It is assumed that very low deformation temperatures will exclude grain growth and hinder the redistribution of dislocations (increasing their density), which should contribute to further refinement of microstructure [55].

Deep cryogenic treatment is considered to be cooling below  $-153\text{ }^{\circ}\text{C}$ , to a temperature below the end of the martensitic transformation, *i.e.*, to the temperature of liquid nitrogen, at a rate below the critical one, holding to complete phase transformations and subsequent heating to normal temperature. Cryogenic cooling is carried out once and does not need to be repeated, since the properties of the material acquired because of hardening and deep cold treatment are preserved throughout the entire service life of the product. The range of cryogenic temperatures from  $0$  to  $-272\text{ }^{\circ}\text{C}$  is associated with polymorphic transformations in the processed material. As the temperature decreases, most materials become stronger and more wear-resistant, and their tensile strength and hardness increase. At a temperature of minus  $196\text{ }^{\circ}\text{C}$  (the boiling point of liquid nitrogen), the tensile strength of most metals is 2–5 times greater than at room temperature; the strength of some plastics and glass increases up to 8 and 12 times, respectively. At a temperature of  $-269\text{ }^{\circ}\text{C}$ , ultimate strength of copper is 2 times greater than at room temperature, and that of aluminium is 4 times greater [1, 20].

Significant number of publications and patents deal with the issues of cryogenic hardening of cutting tools, as a result of which the strength and wear resistance of tool alloyed steel grades increase by 2–5 times [10, 11]. According to Integrated Cryogenic Systems Inc. (Canada), after cryogenic treatment, there are significant improvements in the wear resistance of steels compared to hardening at high temperatures pre-

sented in Ref. [56], while the holding time of products can be in a wide range from several minutes or hours to a day or more. There are no unambiguous recommendations on the choice of cooling modes in publications and depend on specific steel grades, sizes and shapes of products, and previous stages of high-temperature processing. A known method of hardening carbide and diamond drilling tools [42], including its heat treatment by cold immersion in liquid nitrogen, which ensures a consistently high output and drilling speed in various rocks. It should be noted that the effects of ultralow temperatures were observed not only in metals, but also in ceramics and some polymers with a crystalline structure. Maintaining the temperature regime is provided by a special cryogenic installation containing a computer system for controlling the supply of coolant to an isothermal chamber with vacuum thermal insulation. It was considered [55] that the most effective is not a separate application of cryogenic treatment, but its inclusion in the overall process of heat treatment followed by low-temperature tempering. Subsequent heating and holding eliminate the internal stresses that have arisen during cooling due to the difference in the thermal expansion coefficients of austenite and martensite, which is especially important for processing steels with a large initial amount of retained austenite. Therefore, installations are used that can provide subsequent heating of products, as well as cyclically repeated cooling and heating, which reduces the total processing time. In the cold treatment method [54], the cutter is cooled in liquid nitrogen five times, after which it is used no later than two days, when the effect of increased durability is maximum.

Relieving internal stresses and improving wear resistance is especially important for large tools such as dies. During processing, tools are not immersed in liquid nitrogen, but placed in a cryogenic heat exchanger, where they reach a temperature of  $-196\text{ }^{\circ}\text{C}$  in 8 h and are kept at it for 8–20 h. Then, the cooled tools are re-tempered (at  $-149\text{ }^{\circ}\text{C}$ , according to the recommendations of experts) to relieve the stresses caused by the newly formed martensite. The cost of cryogenic processing of tools together with re-annealing is still quite high and reaches \$6–8 for a solid three-tooth end mill and \$3 for a shaped one. Tool factories that carry out mass production of tools, and large machine-building factories, where these tools are then used in large quantities, can buy a cryogenic installation, which costs about \$45 000. The most recommended candidates for this setup are commonly used high-speed steel and carbide tools such as drills, end mills, broaches, reamers, circular and band saws. For dimensional stabilization, standard tiles made of high-alloy alloys can also be subjected to cryogenic treatment [57].

Cryogenic treatment with liquid nitrogen is one of the methods

quite often used in the world practice to increase the hardness and wear resistance of tools, mainly cutting ones, relieve their internal stresses and improve the surface quality. In the study of several materials subjected to cryogenic processing, e.g., when turning low- and high-carbon, bearing steel, titanium alloy and aluminium, it turned out that the workpiece should not be subjected to low-temperature cooling. In this case, the workpiece metal increases its hardness, strength, abrasion in relation to the cutting tool, and preliminary sharp cooling increases the cutting forces arising during processing. Liquid nitrogen should be used to cool a tool (made of high-speed steel or hard alloy), which cryogenic temperature makes harder and more durable. When it was tested for shock loading, it also turned out that the brittleness of the tool material changes insignificantly, and its destruction does not accelerate [43].

Practice has shown that of a number of possible ways to bring liquid nitrogen to the tool, the most effective is its direct supply to the top of the tool, *i.e.*, to the zone of contact with the workpiece. In this case, it is possible to achieve the greatest cooling effect. In this case, the flow of liquid nitrogen is proportional to the amount of heat generated during the cutting process; the workpiece maintains a constant temperature, which prevents the occurrence of dimensional deviations and geometric deformations in it [54].

Cryogenic processing (turning or milling) occurs according to the following principle: jet of liquid nitrogen is fed through a micronozzle located between the front surface of the cutting insert and a chip breaker, which must raise the chip so that it does not interfere with the jet of liquid nitrogen entering the contact zone between tool and workpiece, where the maximum amount of heat is released. Nitrogen, absorbing this heat, quickly evaporates, forming a 'cushion' of gas and liquid between the chips and the surface of the tool, causing such a lubricating effect that no other cryogenic liquid can cause. As a result, secondary chip deformation, coefficient of friction and tool wear are reduced. For additional cooling of the tool, a second additional nozzle can be installed near its tip. At the same time, body of the tool practically does not cool down and retains its strength. Improvement of chip breaking process occurs in three directions: chip embrittlement due to its rapid cooling, improvement of its grain structure due to a decrease in secondary deformation and recrystallization, and improvement of its chip bending. Common problem of build-up on the cutting edge is also eliminated since the rapid cooling in the contact zone reduces the possibility of chip welding. The high pressure of the liquid nitrogen jet contributes to the rapid removal of the build-up, thereby providing the possibility of high-speed processing with obtaining a high surface quality [52].

## **5. Cryogenic Processing of Rolling Equipment Elements**

Cryogenic technology is also used for processing rolls [52]. The advantages of low-temperature treatment of rolls are as follow: increased service life, reduced spots of low hardness, improved wear resistance, achieved dimensional stability, more hardness at greater depths, less susceptibility to surface chipping.

In Ref. [55], an increase in the hardness of rolls material for cold rolling from 64 to 67 HRC (according the Rockwell scale) was obtained due to a decrease in retained austenite from 6–7% to 1.5–1.7%. Considering that, depending on the steel grade and the mode of its heat treatment, austenite content can reach 40%; significant improvement in performance properties can be expected. At the same time, for such large-size products as rolling rolls, issues of optimizing the schemes for supplying and flowing cryogenic coolant play a decisive role. Duration of cryogenic treatment is determined by the geometric dimensions of rolls and required depth of surface layer. Coolant supply regimes can be calculated with sufficient accuracy on the model.

Cryogenic action is used to increase the bending strength and contact endurance of gears [55] operating under increased loads and speeds and restrictions on overall and weight parameters, for example, in aviation and space technology, racing cars. Cryogenic treatment can become an effective means of increasing the resistance of large-sized highly loaded parts of rolling mills: couplings and spindles, gear shafts and gear wheels, where it is difficult to ensure uniform mechanical properties in the manufacture with a diameter of 4000–4500 mm and a width of 500–800 mm of ring gear. Therefore, positive effects are achievement of uniformity of properties, removal of internal stresses and stability of geometric dimensions, which reduces tooth breakage and pitting.

In contrast to known methods of surface hardening (carburizing, nitriding, spraying), low-temperature treatment affects the entire volume of metal (depending on specified cooling mode) and during the entire operating time, *i.e.*, the strength of, for example, teeth or spindle heads does not depend on their contact wear, which can be 2–5 mm or more. Guillotine shear blades or rolls can be re-sharpened many times while retaining the properties acquired after cooling. In addition, cryogenic treatment can also be used in combination with their subsequent surface treatment, which, however, must be carried out after each re-grinding or grinding of parts or tools. Considering that, copper increases its conductivity after low-temperature treatment, this can be considered as one of the ways to save energy in the operation of electric drives of stands with a capacity of 5–12 MW. An important application is to increase the wear resistance of spindles on bronze bearings, which must be replaced after 2–8 weeks. It is expedient to process expensive

large-sized plain and rolling bearings, which experience increased loads and wear due to misalignments and bends of the roll necks, which leads to their sudden failures and damage to the stand equipment [57].

The existing tendency to increase the speed of cold rolling and temper skinning of thin strips and to decrease the thickness of rolled products increases the intensity of thermal processes per unit volume and contact area in the deformation zone. At the same time, requirements for surface quality, geometric accuracy and flatness of wide strips are becoming more and more stringent, especially for applications in the automotive industry and production of household appliances. These contradictions are designed to satisfy new types of cutting fluids (coolants). Traditional natural and synthetic emulsols of various chemical composition and applied concentration limit the resistance of coolant, when it is heated to 150–200 °C in the deformation zone. Most of supplied emulsion is carried away with the strip to subsequent technological operations of annealing, galvanizing, skin pass, which makes it necessary to clean thoroughly the surface, for example, before cladding the strips. In the mass production of strips, ideal cleaning requires additional costs, and, as a rule, is not achieved, which reduces the quality of the resulting coatings and metal strips [54]. During dry tempering of strips with a normalized matte surface, according to the technology requirements, replacement of work rolls is performed after 4–5 rolls due to a decrease in their roughness (3 to 1.5 μm), which limits productivity of existing mills and reduces surface quality stripes. Temperature in the deformation zone also acts as a limitation. The use of coolant to increase skin pass rate is undesirable due to the appearance of contaminants on the surface of strips. Therefore, alternative methods of selective cooling of rolls during skin pass and cold rolling are needed, which are no less effective, but do not leave inclusions on the surface and allow one to control the flatness. The use of cryogenic liquids in rolling production is patented by the largest manufacturer of industrial gases: Air Products and Chemicals Inc. (USA). Cryogenic technology was tested at Sao temper mills Paolo (Brazil) and ThyssenKrupp Steel in Dortmund (Germany) [58], where SMS Demag (Germany) also conducted research on the cold rolling mill of the effects of liquid nitrogen in combination with low volume lubrication technology (low volume lubrication), developed jointly with REBS Zentralschmiertechnik GmbH and involving the formation and supply through the nozzles of an anhydrous mixture of air and emulsol. The use of cryogenic technology in the last stand of cold rolling mills to replace the existing zone cooling system requires some changes in the equipment, which include: introduction of multizone cooling at the inlet of the penultimate stand; additional cooling of strip in the last two spaces between stands; protection of the last stand from the emulsion from the previous stand. Figure 14, *a* [57] shows that liquid



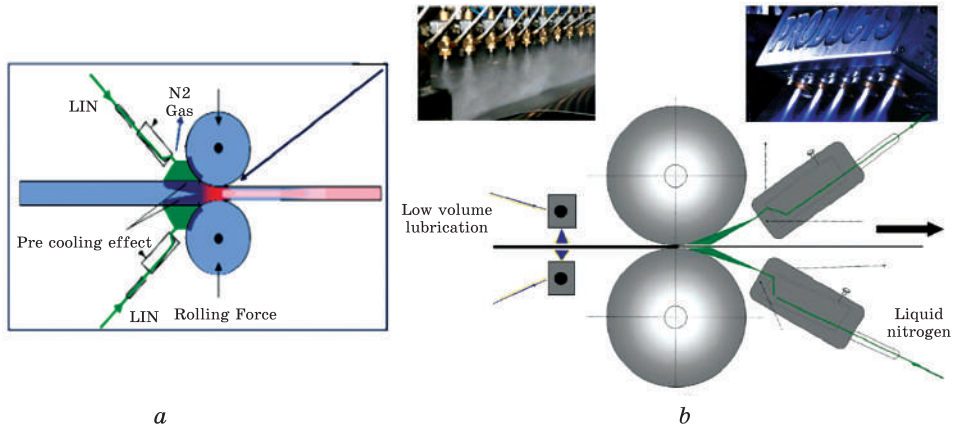


Fig. 14. Schemes of liquid nitrogen supply in the rolling stand on the inlet (a) [57] and output (b) [58] sides

nitrogen is supplied at a temperature from  $-170$  to  $-196$  °C (depending on the pressure) along the width of the strip at the entrance to the deformation zone. Cryogenic cooling of strip and rolls makes it possible to increase rolling speed by 25% and reduce degree of oxidative reactions on the surface of strip when heated during cold rolling (rust spots were eliminated). Along with this, it was found that the supply of liquid nitrogen does not change coefficient of friction in the deformation zone. The supply of a cryogenic coolant at the exit of the stand in Fig. 14, b [58] contributed to a better transfer of roughness from rolls to the strip, which is associated with the contact of backup and work rolls and made it possible to double service life of the work rolls due to a longer preservation of their roughness [60]. Flatness of strip depends on the uniform supply of cryogenic coolant. Therefore, liquid nitrogen can be used for targeted control of the roll profile and elimination of local defects in the strip profile on temper mills that are not equipped with a selective roll cooling system. A method was proposed for using cryogenic liquids and gases to control the profile of a strip and rolls [58], in which the controller regulates the flow rates for each of the nozzles in the line and forms the required contour along the width of the roll. During the stops of the rolling stands, it is necessary to provide for the overlapping of the nozzles to avoid severe overcooling of individual sections of the rolls and disruption of their profile.

## 6. Economic Aspects of Cryogenic Technologies

At large metallurgical plants with converter and blast furnace steel production, liquid nitrogen has a low cost as a by-product in the production of oxygen by air separation by liquefaction. Therefore, at large volumes

of consumption, it is economically expedient to use nitrogen as a coolant. One of the positive factors for conducting research in Dnipro is the presence of Linde Gas Ukraine (Sweden) — the largest Ukrainian and European manufacturer of industrial gases, cryogenic liquids of various degrees of purification and equipment for their storage. Therefore, many problems with the transportation of liquid nitrogen are removed, and its cost as the main consumable for research is reduced. The highest efficiency of cryogenic treatment is achieved by reducing repeated operations of high-temperature hardening. In addition to the rather high price, one of the disadvantages of cryogenic treatment is the film formed after it on the instruments with a thickness of approximately 25 microns. Therefore, tools should be sharpened only after this treatment. Cryogenic processing has not yet reached that stage of technical and economic maturity, which can currently be of mass interest to enterprises in its practical application. However, with the inevitable tightening of environmental laws (regarding machining on machine tools) and increasing competition in the production of cutting tools, above advantages of this processing will undoubtedly attract the attention of many manufacturers and perhaps make them draw practical conclusions. The fundamental limitation on the wide application of low-temperature deformation of metals as a means of improving their physical and mechanical characteristics is the decrease in their plasticity as the temperature of deformation is lowered, as well as the lack of methods for deformation at low temperatures of metals with low plasticity. Abrasive particles located on the periphery of jet act on the treated surface with a force 3–4 times greater compared to liquid drops having the same speed and size. The main problem is the effective injection of abrasive material into a high-pressure liquid jet with the least disturbance of its hydrodynamic characteristics, and then the return of the abrasive from the pulp in the return circuit to increase economic efficiency of the process. Unlike conventional abrasive surface treatment, such as quartz sand, to create abrasive particles, you can use the crystals of the liquid itself, which are formed when it is cooled in a stream. The same method of creating an abrasive is used in high-pressure installations (up to 1000 MPa) for waterjet cutting of metals [57]. Ordinary ice, when cooled to  $-50\text{ }^{\circ}\text{C}$ , approaches and even surpasses some steels in hardness. At the same time, problems of reuse and disposal of used abrasive are being solved. Chilled carbon dioxide can be used for cleaning, or granular dry ice blown with compressed air (dry ice blasting). The effect is achieved both from abrasive action and from local cooling, in which the coating or contaminants are better removed due to the thermal expansion coefficients that are different with the material being cleaned. Manufactured imported manual equipment, *e.g.*, by Triventek (Denmark), is used for cleaning electrical equip-

ment and air ducts and includes installations for the preparation of dry ice. The economy of waterjet cleaning is also determined by the service life of nozzles subject to intense wear. There are many designs of jet heads using highly hard metals and ceramics. When using cryogenic methods of forming an abrasive in a liquid, the wear resistance of the nozzle is increased due to the formation of a small layer of ice on its inner surface, as well as by freezing the liquid after it exits the nozzle [57].

## **7. Safety Precautions with Cryogenic Liquids**

The safety precautions for handling liquid nitrogen should be listed. When using industrial liquid nitrogen [58] with a boiling point of  $-196\text{ }^{\circ}\text{C}$  at atmospheric pressure and oxygen content of up to 3%, it must be considered that when it evaporates, the oxygen content in it constantly increases, since oxygen evaporates at a temperature of  $-183\text{ }^{\circ}\text{C}$ . The resulting nitrogen–oxygen mixture at an oxygen content of more than 30% in contact with organic compounds (lubricant, *etc.*), as well as upon impact, becomes explosive. Therefore, the use of liquid nitrogen with an oxygen content of more than 30% is strictly prohibited. The oxygen content in the mixture is controlled using the Hempel device. Specially trained personnel work at metallurgical enterprises to service cryogenic installations. Rooms in which nitrogen is stored or used must be well ventilated, as an increased concentration of nitrogen (insufficient percentage of oxygen in the air) causes a person to lose orientation, as in oxygen starvation. Nitrogen cylinders or Dewars should not be exposed to excessive heat, and the vessel valves should be opened slowly. When working directly near liquid nitrogen, it is necessary to use special gloves, goggles, and clothing to avoid injuries associated with frostbite of skin areas. Laboplus (Germany) together with Tempshield Inc. (USA) since 1986 offers special protective gloves for industrial use at low temperatures, which protect the wrist, forearm, and elbow. Although the EN511 standard (protective gloves against cold) specifies testing of products at temperatures of  $-50\text{ }^{\circ}\text{C}$ , the manufacturer has tested and recommends their use down to  $-160\text{ }^{\circ}\text{C}$ . The outer material of the gloves is semi-permeable nylon, which according to the manufacturer is 100% water repellent. Cold insulation is provided by a polyolefin/polyester lining and a cotton lining. For long-term work in the gas phase and at the risk of liquid wetting, gloves are equipped with seamless inner gloves, which provides resistance to the penetration of cold through the seam to the fingers [59].

## **8. Conclusions**

At present, direction associated with the use of cryogenic liquids and gases in rolling production and mechanical engineering is developing more and more widely in the world. In the CIS countries, this technology is not yet used in rolling production. In mechanical engineering, low-temperature heat treatment makes it possible to increase the service life of cutting tools by a factor of 2–5 without significant capital expenditures, durability of gear drives of gearboxes, rolling rolls, and other dimensional parts operating under conditions of increased dynamic loads and contact wear [12].

Cooling of cutting tool at low flow rates of cryogenic liquids and high-speed processing of materials makes it possible to improve quality and increase intensity of cutting processes, to refuse or reduce the cost of coolant, cost of its disposal. Quality of plasma spraying of coatings is improved. During hydroabrasive cleaning and cutting of materials, formation of solid crystals as an abrasive in a liquid jet saves on consumption of nozzles and abrasive material and simplifies design of installations [60].

In rolling production, the most promising areas for the use of cryogenic technologies are as follow: descaling and protection of metal from oxidation during hot rolling; acid-free strip surface cleaning before cold rolling and coating; restoration of rolls rough surface during skin pass and in the last stands of cold rolling mills; strip flatness control by selective roll cooling [54].

It is economically feasible to use cryogenic technologies at machine-building enterprises with a large fleet of machine tools and consumption of cutting tools for the production of finished products and in repair shops at large metallurgical enterprises of the CIS, which have their own capacities for the production of liquid nitrogen as a by-product during air separation. However, even with the purchase of liquid nitrogen by small enterprises, its relatively low cost, because high purity is not required, will save on the consumption of spare parts and tools. An alternative is the provision of services for cryogenic processing of products by third-party enterprises that have cryogenic facilities at their disposal [53].

Examples given in the article make it possible to find other industries where, based on cryogenic technologies, it is possible to ensure energy savings, resource saving and safe operation of critical highly loaded machines and units. A more detailed study of the internal processes occurring in structural metals during cryogenic treatment will make it possible to find quickly new areas of application for this type of influence on the properties of materials, the full potential of which has so far been discovered only to a small extent.

Cryogenic cooling is an ecologically friendly solution to traditional emulsion cooling and an effective way to keep the temperature at the phase significantly higher than softening point of the cutting tool material. In theory, this will enhance the processability of hard-to-machine aerospace materials such as titanium and nickel-based super alloys. The main disadvantages of cryogenic cooling are the additional equipment and the high cost of liquid nitrogen. Compared to conventional cooling, however, cryogenic cooling has been noted by many researchers as an excellent opportunity to increase the tool life produced with modified tool inlays that achieve optimized frictional and thermal characteristics. However, some issues of this technology limit its application in real industrial settings rather than on laboratory machines, such as safety concerns regarding the handling of liquid and gaseous nitrogen, cooling of the workpiece, which leads to non-compliance with tolerances on through passage dimensions and the size of the cryogenic plant to gain access to inner diameters. As previously mentioned, this may be the preferred technology in sectors where these restrictions are not valid or are less significantly [1, 60].

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#### **ЗАСТОСУВАННЯ КРІОГЕННИХ ТЕХНОЛОГІЙ У ДЕФОРМАЦІЙНОМУ ОБРОБЛЕННІ МЕТАЛІВ**

Оглянуто застосування кріогенних технологій у процесах термічного та деформаційного оброблень металів. Визначено найефективніші області застосування кріогенних рідин і газів для термооброблення робочих інструментів і металів (титанових, алюмінієвих і мідних стопів) у вальцювальному виробництві та важкому машинобудуванні з метою поліпшення якості продукції, стійкості устаткування й інструменту, пониження екологічного впливу на довкілля та працюючий персонал. Розглянуто вплив кріогенного оброблення й охолодження на термін служби інструменту, його стійкість, зношення, температуру різання, шерсткість поверхні, точність розмірів і сили різання. У результаті застосування кріогенного оброблення та кріогенного охолодження в процесах механічного оброблення збільшується термін служби інструменту та поліпшується шерсткість поверхні, а також понижуються температура оброблюваної поверхні, споживання енергії під час роботи; таким чином зменшується зношення інструменту, що сприяє збільшенню продуктивності. Розглянуто також можливість одержання та зміни наноструктури металу за допомогою кріогенного охолодження. Тематика може бути цікавою дослідникам і вченим у галузі металургії, матеріалознавства та нанотехнологій.

**Ключові слова:** кріогенне охолодження, рідкий азот, стійкість інструменту, шерсткість поверхні, низькотемпературне оброблення, наноструктурні матеріали.