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CRYOGENIC COOLING EFFECT ON THE CHANGE IN THE MICROSTRUCTURE OF METALS

The study of cryogenic treatment of metals has been the subject of a large number of scientific publications; therefore, the preparation of a review article aimed at summarising the current state of knowledge and identifying directions for future research is highly relevant. Although there are several reviews on the cryogenic treatment of tool steels, to date, there has been no comprehensive review addressing the effect of cryogenic treatment on changes in the microstructure of metals. Therefore, in addition, the influence of individual processing parameters, their sequence, and the effect of stabilisation at room temperature on microstructure evolution are examined here in detail. Cryogenic processing of materials is known to enhance properties such as hardness, strength, wear resistance, tensile strength, dimensional stability, corrosion resistance, *etc*. However, the extent of property improvement for cryogenically treated materials reported in the literature is diverse and, in some cases, contradictory. In the present study, an attempt is made to provide a comprehensive review of various scientific publications available in the literature on this topic.

Keywords: microstructure of metals, metals processing, cooling, cryogenic treatment.

1. Introduction

Heat treatments required to obtain the desired properties of various materials are generally well established [1–5]. However, research aimed at improving the mechanical properties and wear resistance of metals through cryogenic treatment is a relatively new field. Cryogenic treatment is commercially applied in various sectors, such as the tooling industry, firearms manufacturing, acoustic equipment, and others. Researchers have focused

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on specific mechanisms, through which cryogenic treatment provides the reported improvements; nevertheless, the underlying mechanisms are not yet fully understood. The concept of enhancing the mechanical properties of steel materials by exposing them to a cold environment has existed for a long time. It is often stated that Swiss watchmakers buried components in the ground to improve the properties of parts used in their watches. In addition, tool manufacturers are known to store tool materials in refrigerated chambers for a certain period in order to enhance their performance.

Cryogenic treatment (CT) is a process in which the material is slowly cooled to −196°C, held at this temperature for a specified duration, subsequently heated in a controlled manner to room temperature, and then tempered. It is recommended as a supplementary process to conventional heat treatments. The most well-known mechanism in steels is the transformation of retained austenite remaining after quenching into martensite. Another important mechanism, also illustrated using steels, is lattice contraction at low temperatures, which promotes the diffusion of carbon atoms out of the lattice and their interaction with carbide-forming elements, resulting in the formation of very fine carbides.

Many studies emphasise that cryogenic treatment modifies not only the surface properties of a material but also its internal structure [6–8]. Since the process is carried out at extremely low temperatures, the treatment duration, especially, for large components, is relatively long. There are numerous non-academic reports and promotional materials claiming that cryogenic treatment enhances material properties. It is known that this treatment affects a wide range of metals, including steel, aluminium, and copper. Cryogenic heat treatment does not replace conventional heat treatments but rather serves as a complementary process. The liquid nitrogen used in the process and its application must be handled with care, and, whenever possible, direct contact between liquid nitrogen and the material surface should be avoided.

Liquid nitrogen is the most preferred cryogen in cryogenic cooling technologies [9]. Nitrogen (N_2), with a boiling point of −195.8°C, is the most abundant gas in the atmosphere (78.03%) [10]. Since it is lighter than air, it tends to evaporate [11] and disperse during cryogenic cooling. For this reason, when existing cryogenic cooling systems and methods are compared, liquid nitrogen is regarded as environmentally friendly, non-hazardous to human health, and a means of enhancing manufacturing sustainability. In addition to the fact that cryogenic cooling has been applied in various manufacturing processes since the 1950s [11] and is considered one of the environmentally benign manufacturing methods, it has been reported to extend cutting tool life [12], provide better surface quality [13], reduce subsurface microstructural deformation generated after machining [14] and white layer formation [15], and, in some studies, reduce shear forces [16], among other benefits.

Among cryogenic cooling media, liquid nitrogen (LN_2) is the most widely used; however, studies employing carbon dioxide (CO_2) are also frequently reported in the literature [17, 18]. Some researchers [19] argue that cryogenic cooling using LN_2 at very low temperatures (–196°C) adversely affects the mechanical and physical properties of the work piece and, therefore, suggest the use of CO_2, which has a higher boiling point (–78.5°C) than LN_2. Others [14] note that, when CO_2 is used, it is difficult to control the formation of dry ice, which is the solid phase of CO_2, and that, because CO_2 is heavier than air, its use may lead to oxygen deficiency for workers and additionally contribute to the greenhouse effect, thereby negatively affecting global warming. Furthermore, they emphasise that LN_2 is lighter than air and, therefore, evaporates and disperses into the atmosphere, is the most abundant atmospheric gas, and has a lower boiling point than CO_2, which constitutes a significant advantage. In addition to these aspects, several important considerations must be taken into account when using LN_2. In manufacturing processes involving LN_2, adequate ventilation of the working environment must be ensured to prevent oxygen deficiency caused by a reduction in oxygen concentration due to LN_2 use. Moreover, a safety relief valve must be installed to maintain an appropriate pressure balance in cryogenic cooling equipment operating under high pressure as a result of the high expansion ratio of LN_2 (1:693). Cryogenic cooling systems should also be equipped with appropriate protective devices to prevent exposure to factors that may pose occupational health and safety risks at cryogenic temperatures [20].

In recent years, hybrid cooling/lubrication methods have gained significant importance, aiming to provide not only effective cooling but also sufficient lubrication. Minimum quantity lubrication (MQL), used as an economical and environmentally friendly method, is another approach that enhances manufacturing sustainability by reducing friction and temperature in the cutting zone [21]. Hybrid cooling systems combining MQL with LN_2 [22] or CO_2 [21, 22] have been applied in various manufacturing processes in order to improve lubrication performance in addition to the effective cooling capability of cryogenic cooling.

2. Application of Cryogenic Cooling in Machining Processes

Nitrogen is used in manufacturing in two different ways: cryogenic treatment and cryogenic cooling [23]. The former is a type of heat treatment applied to improve the mechanical and physical properties of various engineering materials. It is carried out by gradually cooling materials to cryogenic temperatures, holding them at these temperatures for an extended soaking period (*e.g.*, 24 h), and then slowly restoring the temperature to room temperature [24]. The latter is applied to engineering materials that experience high-temperature-related problems, particularly during machin-

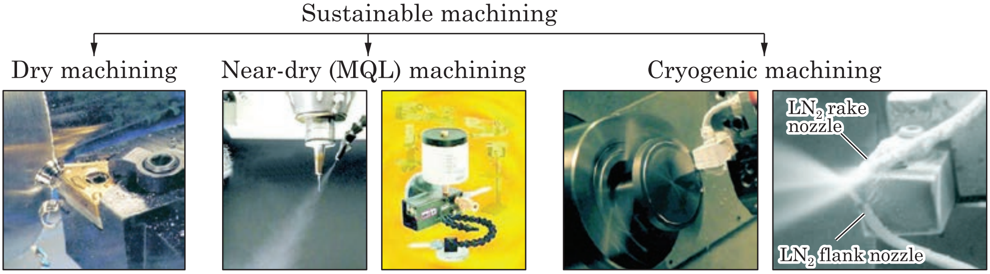


Fig. 1. Sustainable manufacturing methods [33]

ing, such as superalloys [25], titanium alloys [9], stainless steels [26], and hardened steels [27]. Owing to its effective cooling capability, cryogenic cooling allows higher cutting speeds and material removal rates, extends tool life, and improves surface quality. At present, cryogenic cooling is considered a preferred productivity-enhancing and environmentally friendly approach in machining and is applied to a wide range of engineering materials in addition to conventional chip-removal operations, such as turning [26], milling [14], drilling [28], reaming [29], grinding [30], polishing [31], and electrical machining [32], among others. In this method, the cryogenic fluid is applied directly or indirectly to the work piece, the cutting zone, the cutting tool, or various combinations thereof using different delivery strategies. Such systems are employed for cryogenic cooling in turning, milling, drilling, and related processes. As an alternative to conventional cutting fluids used for cooling and/or lubrication in machining operations, especially, those, in which high temperatures arise without mechanical contact, cryogenic fluids are applied indirectly through various application methods, to create a sustainable manufacturing environment and enhance machining performance.

Considering current research, numerous studies compare the performance of cryogenic cooling in various manufacturing processes with dry machining, conventional cutting fluids, minimum quantity lubrication, and hybrid cooling/lubrication systems. Figure 1 [33] illustrates the four main methods used in sustainable manufacturing.

Cryogenic cooling, as part of sustainable manufacturing approaches, outperforms dry machining due to its effective cooling capability. Owing to their lubrication characteristics, hybrid cooling/lubrication strategies have gained significant importance in recent years, particularly, when combined with cryogenic cooling.

In current studies, cryogenic cooling is generally described as one of the methods for enhancing machinability and promoting environmentally friendly manufacturing. However, in order to achieve optimal results using this approach, cryogens must be applied through different delivery strategies tailored to the specific manufacturing process. This is because diffe-

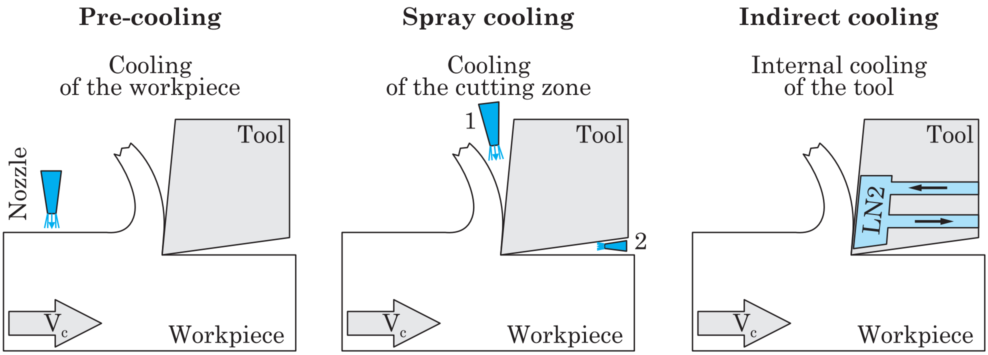


Fig. 2. Cryogenic cooling methods [34]

rent machining processes involve distinct chip formation mechanisms, as well as variations in work piece and cutting tool materials, which respond differently under cryogenic cooling conditions.

Figure 2 [34] schematically illustrates the different cryogenic cooling methods.

As shown, cryogenic cooling applications are implemented using work piece cooling with pre-cooling, cutting zone cooling *via* spray application, and indirect cooling of the cutting tool. The work piece cooling method involves applying a cryogenic bath by immersing the work piece in the cryogen or spraying the cryogen onto the work piece immediately before chip removal during machining [23]. Pre-cooling the work piece using a cryogenic bath improves the machinability of highly elastic and adhesive materials, such as viscoelastic polymers [35]. Spraying cryogen onto the work piece before machining also renders the chips formed during cutting brittle, as the cryogen penetrates the material, thereby, facilitating chip formation, when machining ductile materials. Cutting zone cooling can be achieved through two main approaches: spraying cryogen into the cutting zone using one or multiple external nozzles [13, 14, 36], or delivering cryogen internally through the insert [26, 37] or the tool holder [38]. The primary goal of this method is to reduce the temperature in the cutting zone to minimize the chemical interaction of the cutting tool with the work piece material, thereby reducing adhesion and diffusion, improving machining performance, and preventing work piece damage that may occur due to high temperatures [23]. In indirect cooling, the cryogen passes only through the cutting tool to cool the tool and, indirectly, the cutting zone or work piece. In this method, the cryogen reduces the cutting zone temperature and evaporates [32]. Since the cryogen does not come into direct contact with the work piece, negative effects such as dimensional variations caused by cryogenic cooling are avoided [39]. However, the effectiveness of this cryogenic cooling method largely depends on the properties of the cutting tool material [23]. Indirect cryogenic cooling methods are also applied

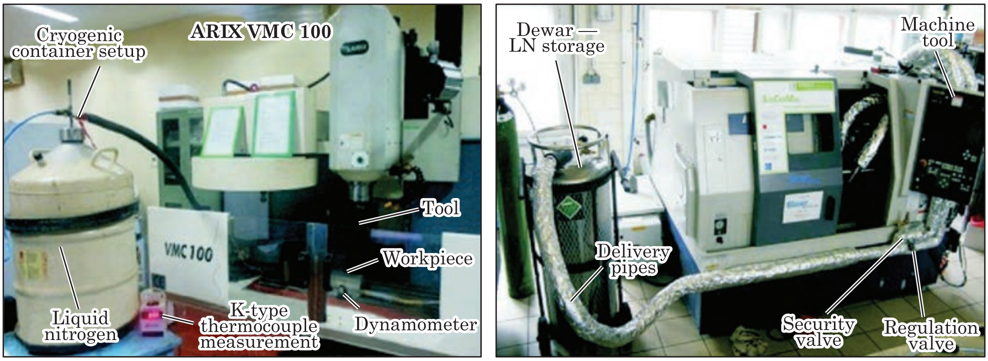


Fig. 3. Cryogenic cooling systems used in manufacturing processes: (*a*) compressed using a compressor [36], (*b*) self-pressurised cryogenic systems [20]

in non-contact manufacturing processes, such as electrical discharge machining (EDM) [32], to enhance performance.

Considering all these methods, the most economical and effective way to select an appropriate cryogenic cooling method for a material in any manufacturing process is to deliver a sufficient amount of cryogen to the precise location using the suitable method, depending on the processing conditions [40]. In a study on cryogenic turning of Ti_6Al_4V alloy using various cooling approaches [40], the cooling effectiveness ranged from poor to excellent as follows: dry machining, indirect cooling of the cutting tool with cryogen, conventional cutting fluid cooling, pre-cooling of the work piece with cryogen, cryogenic cooling of the tools' flank surface, cryogenic cooling of the tools' rake surface, and simultaneous cryogenic cooling of both the flank and rake surfaces. Efficient cooling of the cutting zone significantly enhanced the machinability of this alloy. Moreover, cryogenic cooling affects material properties and the coefficient of friction between the cutting tool and work piece. Depending on the work piece and tool material used, it can either improve or reduce the lubricating effect [41]. For this reason, it is critically important to apply a cryogenic cooling method that enables optimal results, taking into account the specific manufacturing process and the materials of the cutting tool and work piece.

Cryogenic cooling systems used in manufacturing processes can be obtained as ready-to-use commercial equipment [31] or employed by researchers in the form of compressors to increase the pressure of the cryogenic flow [36] or self-pressurised cryogenic tanks [42]. Figure 3 illustrates various applications of cryogenic equipment. In cryogenic cooling systems, cryogens can be applied by researchers at different pressures and flow rates.

Cutting forces generated during machining processes directly affect energy consumption and overall production costs. Under cryogenic cooling, the work piece material becomes brittle at low temperatures, and an increase in its strength is observed, which can lead to higher cutting forces

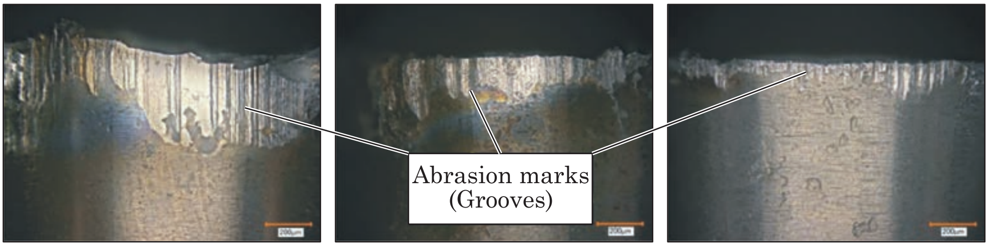


Fig. 4. Comparison of flank wear after 5 min of machining a NiTi shape-memory alloy under different machining conditions [47]

[43]. On the other hand, the literature also reports that cryogenic cooling has a lubricating effect, resulting in reduced cutting forces [44]. The impact of cryogenic cooling varies significantly depending on the work piece and cutting tool materials used in the manufacturing process, as well as the cooling technique employed. Therefore, it cannot be stated categorically that cryogenic cooling universally increases or decreases cutting forces [23]. Studies have shown that cryogenic cooling in different manufacturing processes can either increase [14, 37, 43] or decrease [12, 36, 43] cutting forces. Furthermore, it has been suggested that any reduction in tool life due to increased cutting forces can be mitigated by modifying the tool geometry to accommodate cryogenic conditions [45].

Tool wear leads to the loss of the cutting tools' original geometry, production downtime, reduced machining efficiency, and deterioration of surface quality. Cryogenic cooling, particularly in the machining of difficult-to-machine engineering materials such as titanium alloys and nickel-based superalloys, which exhibit low thermal conductivity and high chemical reactivity with cutting tools, is one of the methods used to reduce high temperatures in the cutting zone, improve machinability, increase material removal rates, and extend tool life. In a study on Ti_6Al_4V machining [38], this effect was attributed to cryogenic cooling producing shorter chips, which reduces friction-induced heat and shortens the contact length between the tool and the chip, thereby, decreasing the amount of heat transferred to the cutting tool. Additionally, cryogenic cooling lowers high temperatures in the cutting zone, reducing thermal interaction between the cutting tool and work piece, which diminishes adhesion and friction tendencies [46]. Figure 4 [47] illustrates the wear mechanisms observed after 5 minutes of machining a NiTi shape-memory alloy under dry, MQL, and cryogenic conditions. The beneficial effect of cryogenic cooling, achieved by simultaneously applying LN_2 to both the rake and flank surfaces of the cutting tool, on tool wear under these conditions is evident. Cryogenic cooling has been reported to improve tool wear not only in turning but also in other manufacturing processes, such as milling [46], drilling [36], and electrical discharge machining [32]. However, despite studies [37] indicating

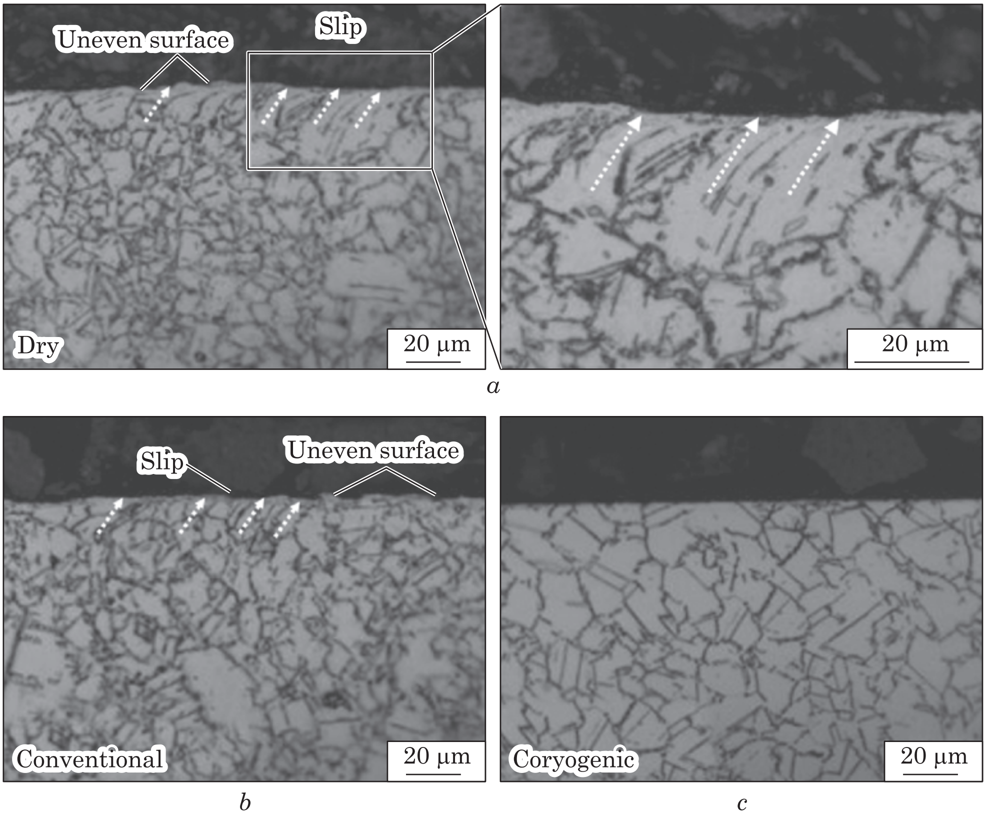


Fig. 5. Microstructure images of the machined subsurface of Inconel 718 under different machining conditions [14]

that cryogenic conditions can sometimes reduce tool life, other research [22] suggests that hybrid cooling/lubrication methods are more effective in enhancing tool wear performance than cryogenic cooling alone.

Surface integrity criteria, including surface roughness, hardness, residual stresses, and subsurface microstructural changes, determine the service life of the final product [25]. Studies on the effect of cryogenic cooling on surface integrity across various manufacturing processes have reported that, compared to conventional cooling conditions, it improves surface roughness [48], reduces dimensional deviations [37], and decreases tensile residual stresses and surface cracks generated during machining [49]. Figure 5 shows the microstructural changes beneath the machined surface of Inconel 718 after milling under different cooling environments. High machining temperatures caused deformation of the grain structure. In contrast, experiments conducted under cryogenic conditions demonstrated reduced plastic deformation and improved surface roughness. Penetration of LN_2 into the cutting zone enhanced surface integrity [14].

In studies focusing on the machinability of titanium alloys through cryogenic treatment, it is evident that most research has concentrated on EDM. Rupinder Singh and colleagues subjected commercially pure titanium (Titan 15, ASTM Gr.2) to cryogenic treatment at –80 °C for approximately 20 hours [50]. The materials' machinability was then evaluated using EDM. The experimental design was based on the Taguchi method, and the outcomes measured included material removal rate (MRR), tool wear rate (TWR), surface roughness, and dimensional accuracy. Cryogenic treatment resulted in improvements of 60.39%, 58.77%, 7.99%, and 80.00% in these parameters, respectively [51]. In another study, Gill and colleagues subjected Ti_{6246} to cryogenic treatment at –196 °C for 24 hours, followed by EDM drilling. Blind holes with a diameter of 10 mm were machined using copper electrodes. The results showed that cryogenic treatment enhanced MRR, increased the effective wear rate (material removal rate per tool wear), and reduced TWR. Additionally, samples subjected to deep cryogenic treatment exhibited improved dimensional and positional tolerances of the holes [52]. Jatti and colleagues also performed cryogenic treatment on NiTi shape-memory alloy at –185 °C for 24 hours. The study examined the electrical conductivity of the NiTi alloy and experimentally confirmed that cryogenic treatment significantly increased its conductivity. The material removal rate was improved by approximately 19% [53]. In another study on shape memory alloys, NiTi alloys were cryogenically treated for 3 hours, after which microhardness and phase analyses were conducted. The results indicated that cryogenic treatment increased microhardness without causing significant changes in the phase composition [54].

In addition to these studies, Kumar and colleagues investigated the effect of EDM on cryogenically treated $Ti_5Al_{2.5}Sn$ alloy using various machining parameters [55]. One group of alpha-alloy specimens underwent shallow cryogenic treatment at –110 °C for 24 hours, while another group was subjected to deep cryogenic treatment at −184 °C. The results were evaluated based on machining performance, MRR, microhardness, tool wear, and surface roughness. The study found that specimens subjected to both deep and shallow cryogenic treatments exhibited significant improvements in performance compared to untreated samples. Different EDM parameters yielded varying results regarding the effect of cryogenic treatment [55]. According to the study, the impact of cryogenic treatment was more pronounced under relatively aggressive machining conditions, whereas under moderate machining parameters, the difference was less noticeable [55]. Based on the literature, it has been concluded that improving the machinability of titanium alloys is possible by addressing factors such as low thermal conductivity, susceptibility to friction and adhesion, irregular internal structure, and the presence of residual stresses in the material, all of which typically hinder machinability.

An analysis of the literature on cryogenic treatment and other titanium-related studies shows that these investigations primarily focus on the wear testing of titanium alloys under cryogenic conditions [56, 57] and on manufacturing processes utilizing cryogenic agents, such as liquid nitrogen and dry ice [58, 59].

3. The Effect of Cryogenic Cooling on Microstructural Changes in Metals

Metal pressure treatment has a significant effect on changing the microstructure of metals [60–69]. The use of cryogenic cooling further enhances this effect. A study conducted on a copper alloy ($Cu_{76.12}Al_{23.88}$) investigated the effect of cryogenic treatment at temperatures ranging from 25 to 600 °C on thermal diffusivity, specific heat capacity, thermal conductivity, and the coefficient of thermal expansion. In this study, copper samples were heated to 800 °C and then subjected to deep cryogenic treatment for 10 minutes. The results indicated that cryogenic treatment increased thermal diffusivity, thermal conductivity, and the coefficient of thermal expansion, while having no significant effect on other properties [70]. Similar findings were reported in other studies [71]. In addition, other research has shown that cryogenic treatment enhances the performance of copper electrodes used in spot welding [72].

In the studies [72, 73], a technology was tested for commercially pure copper that combined quenching and cold plastic deformation using the equal channel angular pressing (ECAP) method with intensive cooling in liquid nitrogen. This treatment resulted in a homogeneous fine-grained structure with a predominance of high-angle grain boundaries and improved mechanical properties of copper. A distinctive feature of the microstructure after ECAP at cryogenic temperatures was the formation of dislocation clusters. In contrast, ECAP conducted at room temperature showed a lower dislocation density and more clearly defined grain boundaries (Fig. 6). With intensive nitrogen cooling, the ultimate tensile strength of copper increased by 140 MPa, the yield strength reached 175 MPa, the elongation decreased by 2%, and the reduction in area increased by 2% compared to the initial values. Intensive nitrogen cooling after each ECAP pass contributes to significant strengthening of copper by suppressing post-dynamic recrystallization and maintaining a high dislocation density. It also promotes the activation of mechanical twinning, which serves as an additional mechanism of plastic deformation.

Another widely used group of materials subjected to cryogenic treatment is WC–Co cutting inserts. In a study in this field, the effects of both superficial and deep cryogenic treatments on tungsten carbide (WC–Co) inserts were investigated, with mechanisms of treatment explained. The internal structures resulting from superficial and deep cryogenic treatments were analyzed using high-resolution transmission electron microscopy (HRTEM) and scanning electron microscopy (SEM), but no visible

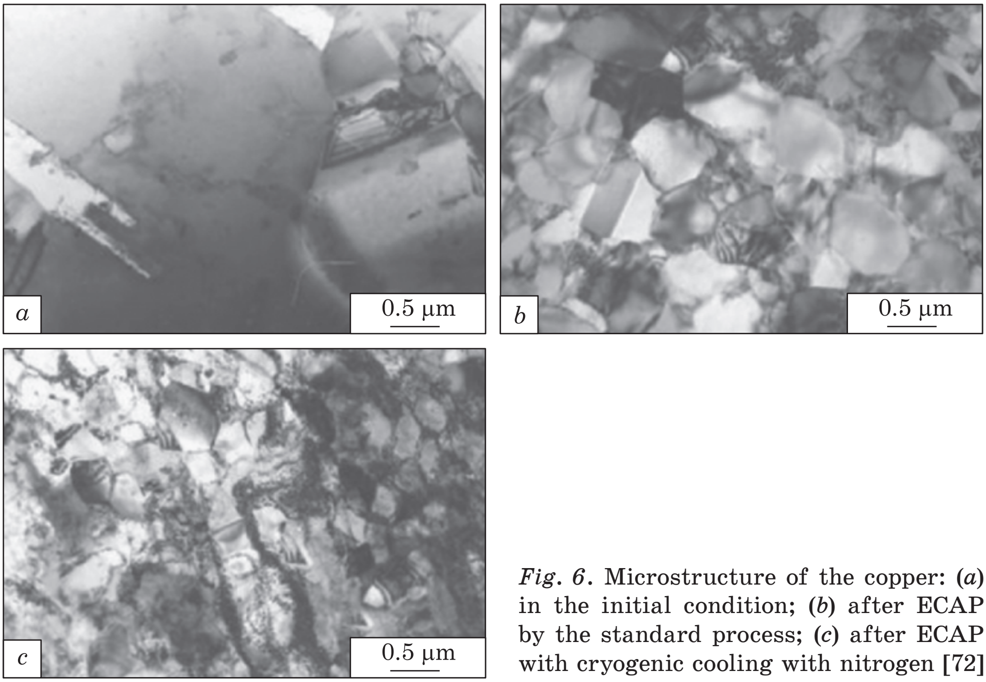


Fig. 6. Microstructure of the copper: (*a*) in the initial condition; (*b*) after ECAP by the standard process; (*c*) after ECAP with cryogenic cooling with nitrogen [72]

changes in microstructure were observed. However, x-ray diffraction (XRD) analysis revealed that the cobalt binder in the carbide structure underwent martensitic transformation as a result of cryogenic treatment. After selectively dissolving tungsten from the surface *via* electrochemical methods, XRD patterns were analysed using the Rietveld method, showing that the fraction of ε-Co after deep cryogenic treatment was higher than after shallow cryogenic treatment and in the untreated sample. Additionally, it was observed that subsequent annealing reduced the amount of martensitic transformation induced by cryogenic treatment [74].

In another study, WC/Fe/Ni carbide material underwent deep cryogenic treatment for 2, 12, and 24 hours. Selective electrolytic corrosion tests were performed to observe the effects of cryogenic treatment. Internal friction, mechanical, wear-resistant, and corrosion properties of the material were investigated. The results showed that the binder structure (Fe–Ni) underwent martensitic transformation, while tungsten precipitated within the structure. It was also reported that both the wear coefficient and friction coefficient significantly decreased, although fracture toughness showed a slight reduction [75].

In the study [76, 77], metallographic analysis of the microstructure of an aluminium alloy after ECAP in a matrix with parallel channels at room temperature and with liquid nitrogen cooling showed that highly deformed grains/subgrains formed in both cases. However, in experiments with ad-

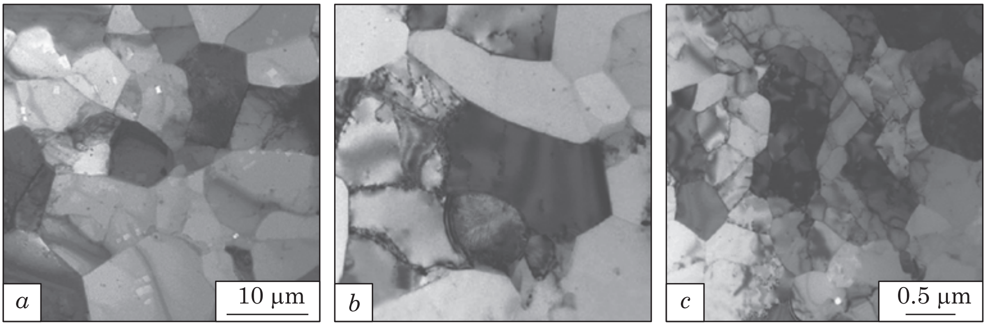
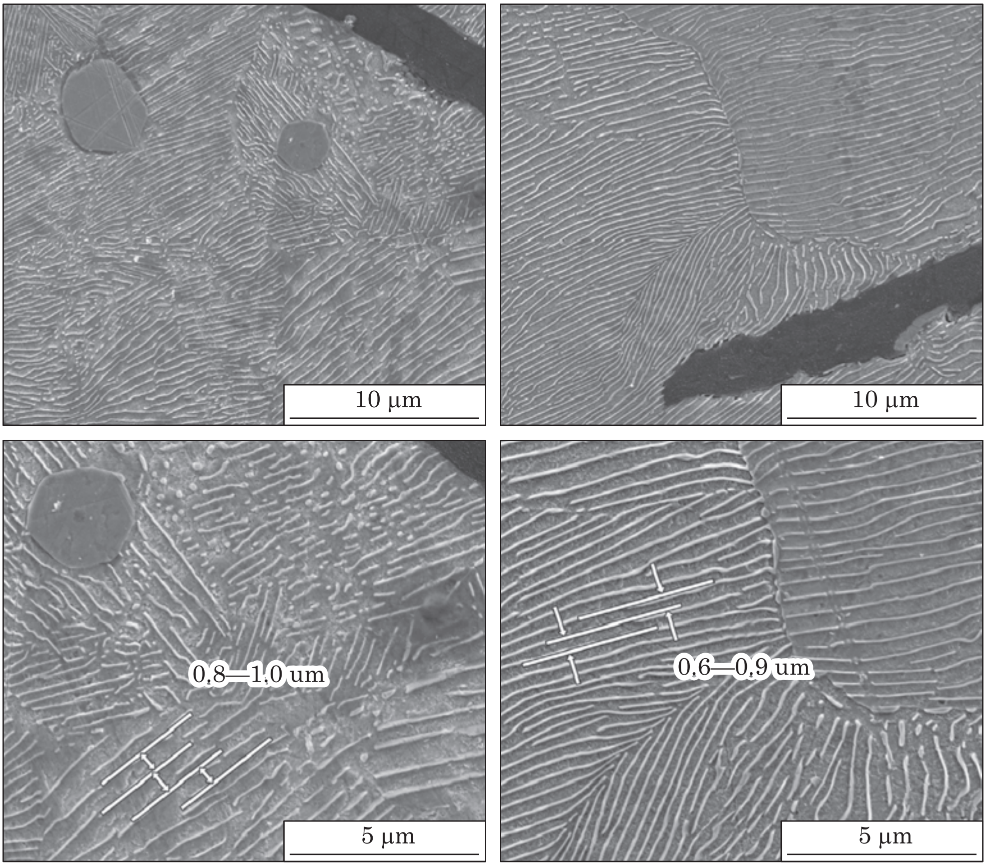


Fig. 7. Microstructure of aluminium alloy D16 (TEM): (*a*) initial state (quenching from 500 °C in water); (*b*), (*c*) after four passes of standard ECAP and with cooling in liquid nitrogen, respectively [76]

ditional liquid nitrogen cooling after ECAP, a finer and more dispersed structure with smaller grain size was observed (Fig. 7), as cryogenic treatment suppresses spontaneous (metadynamic) recrystallization in the alloy. During nitrogen cooling after ECAP, dislocation motion slows down, reducing the intensity of dynamic recovery. This leads to an increase in dislocation density up to $\rho = 6 \cdot 10^{14}$ m^{-2}. Consequently, the microstructure obtained after ECAP with nitrogen exhibits diffuse, non-equilibrium, and poorly defined grain boundaries, which is consistent with the findings of [78]. The alloy structure also contains dislocation cells and dense dislocation walls. In contrast, ECAP at room temperature results in a microstructure with a lower dislocation density ($\rho = 4 \cdot 10^{14}$ m^{-2}) and more clearly defined grain boundaries.

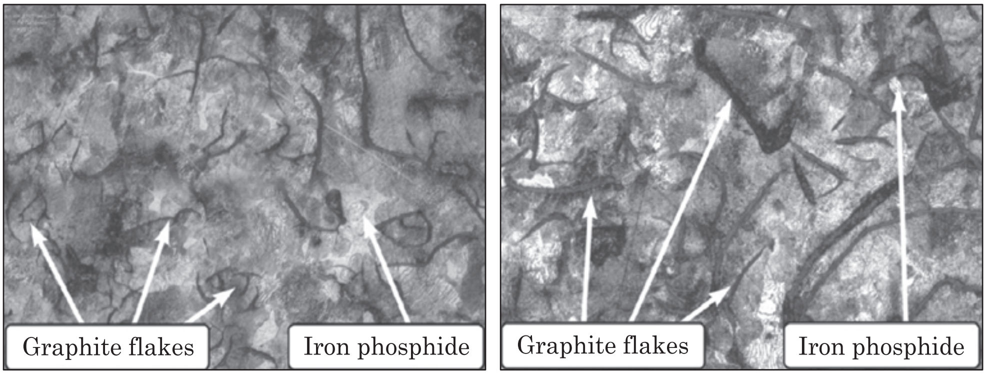
Grey cast iron consists of graphite flakes embedded in a matrix primarily composed of ferrite, pearlite, and/or martensite. In addition to the matrix characteristics, the size and distribution of the graphite flakes strongly influence the materials' properties [79]. It has been found that CT affects the microstructure of pearlitic cast iron by refining the pearlitic matrix and reducing the interlamellar spacing [80]. Furthermore, CT promotes the formation of long, thin, and continuous graphite flakes (Fig. 8). While CT led to a slight increase in hardness (1.5%), wear resistance showed mixed behaviour [80]. Wear resistance increased under high contact pressure but decreased under low contact pressure.

In another study, Slatter *et al.* [81] found that CT increases the impact wear resistance of lamellar (flake) graphite cast iron by changing the failure mechanism from brittle fracture to plastic deformation. Since optical microscopy did not reveal significant microstructural changes, high-resolution methods such as SEM/TEM can uncover the mechanisms underlying the improvement [81] (Fig. 9). In the study [82], cast iron with a martensitic microstructure containing dispersed vanadium carbides was subjected to CT. It was found that, in addition to the transformation of retained austen-



Non-cryotreated Cryotreated

Fig. 8. Electron micrographs of cast iron before and after cryogenic treatment [80]



a *b*

Fig. 9. Microstructures of untreated lamellar (flake) graphite cast iron (*a*) and of lamellar (flake) graphite cast iron subjected to cryogenic treatment (*b*) [81

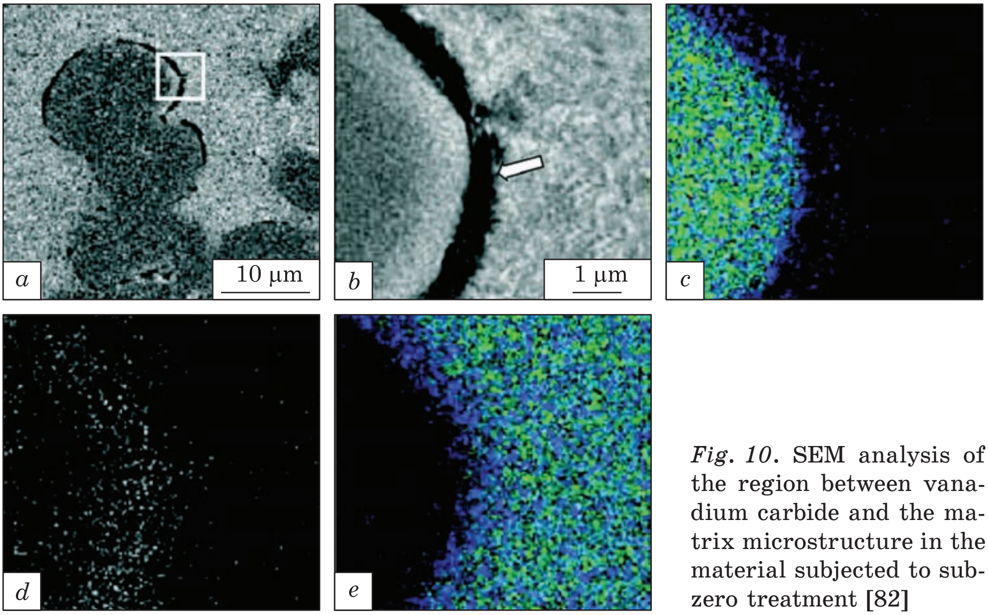


Fig. 10. SEM analysis of the region between vanadium carbide and the matrix microstructure in the material subjected to subzero treatment [82]

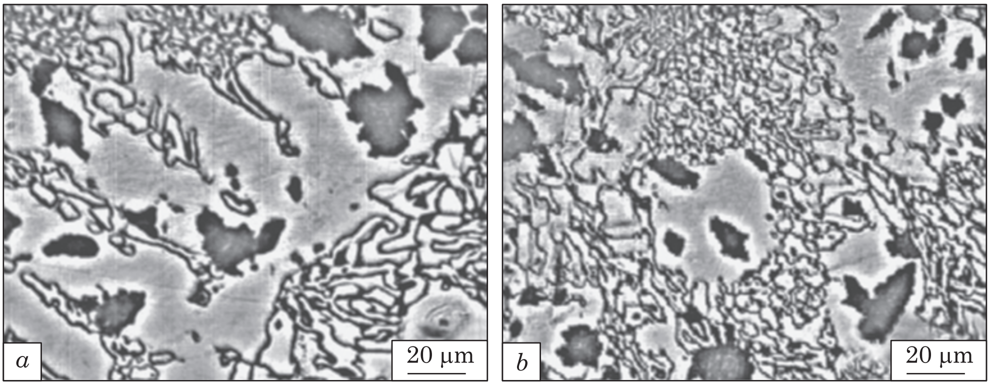


Fig. 11. The microstructure of the high-chromium cast iron: (*a*) air cooling after subcritical treatment at 550 °C and (*b*) cryogenic treatment after subcritical treatment at 550 °C [85]

ite into martensite, a carbon layer forms over the vanadium carbides, which reduces the bonding between the carbides and the matrix (Fig. 10) [82].

While CT enhances the precipitation of secondary carbides, the eutectic carbides remain unchanged. A uniform distribution of fine secondary carbides within the martensitic matrix significantly increases the bulk hardness and resistance to abrasive wear. Due to the dispersion strengthening effect, the fine secondary carbides enhance the strength of the matrix, which in turn provides support to the carbides, preventing chipping and groove formation during abrasive wear [83]. Even if complete transfor-

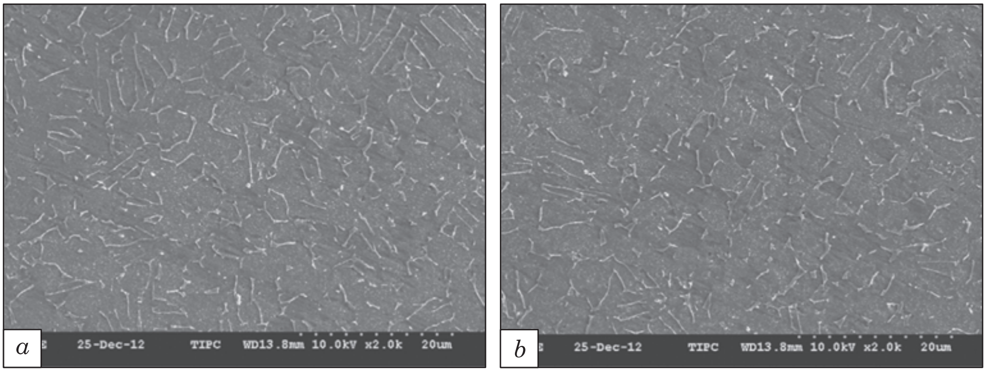


Fig. 12. SEM micrographs of (*a*) untreated and (*b*) deeply-cryogenic-treated samples [89]

mation of retained austenite is not achievable, its presence in minimal amounts is beneficial, as it imparts microplasticity to the matrix, preventing crack initiation and propagation [84]. In addition to the transformation of retained austenite and carbide precipitation, the grain-strengthening effect due to the refinement of the martensitic matrix during CT also contributes to improved wear resistance [85] (Fig. 11).

Soaking time is the second most important parameter after soaking temperature that affects the enhancement of properties in materials subjected to CT [86–99]. The total duration of the CT cycle largely depends on the soaking time. Accordingly, changes in soaking time have a significant impact on the overall cost of CT (Fig. 12).

Gordon *et al.* [97] investigated different soaking times at 83 K and found that soaking time did not affect the transformation of retained austenite into martensite. Popandopulo *et al.* [98] used direct immersion in liquid nitrogen for 15 minutes. Zhmud [100] observed no significant changes in the service life of tool steel when the soaking time varied from 5 minutes to 10 hours; however, it was noted that prolonged soaking time could negatively influence tool life. Berlien [101] recommended 6 hours for the combined phase of cooling and soaking. Barron *et al.* [102] tested soaking times ranging from 1 minute to 24 hours for T8 steel and identified 24 hours as the optimal soaking time for achieving the best results. Later, Barron [103] used a 24-hour soaking period to study the effect of CT on other high-speed steels (HSS). A soaking time of 24 hours at 77 K has also been suggested by several other researchers for HSS. Yun *et al.* [104] investigated soaking times of 24 and 48 hours at 77 K and concluded that a 48-hour soaking time increased both room-temperature hardness and red hardness. Mohan *et al.* [105] studied the effect of soaking times of 6 and 24 hours at different soaking temperatures. A 24-hour soaking time at 163 K provided better results compared to 6 hours at 93 K, and they noted that soaking time was more important than soaking temperature. Dhokey *et al.*

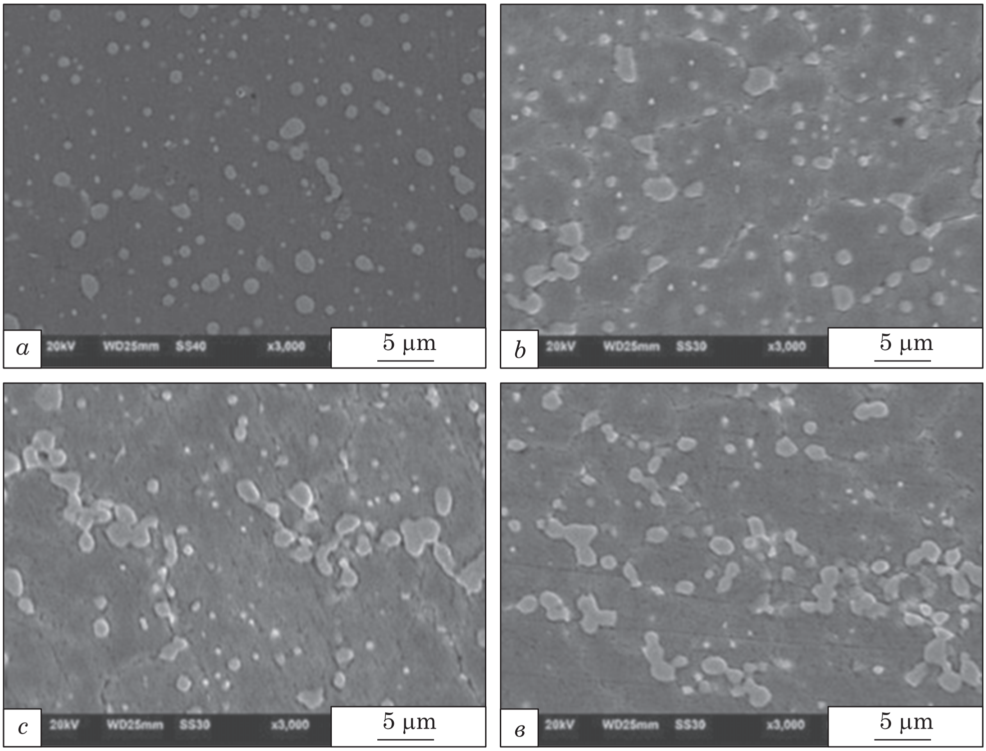


Fig. 13. Microstructure of M2 steel after cryogenic treatment at different soaking times: (*a*) 1 h, (*b*) 4 h, (*c*) 12 h, (*d*) 24 h [108]

[106] conducted experiments with varying soaking times (16, 24, 32, and 48 hours) in terms of residual compressive stresses and found minimal residual stresses at a 24-hour soaking period. A soaking time of 168 hours was proposed by Huang *et al.* [107] for M2 steel, which differs significantly from recommendations by other authors. Li *et al.* [108] investigated the effect of soaking time on the size and distribution of carbides in M2 steel. The corresponding microstructure is shown in Fig. 13. The micrographs clearly indicate that fine-grain precipitation continues during the 24-hour soaking period, despite the growth of secondary carbides. The effect of soaking time on D2, Vanadis 4, and H13 steels was also studied [109–111]. It was found that soaking time does not affect the hardness of D2 and Vanadis 4 steels; however, the hardness of H13 steel improved at a soaking time of 400 minutes. Das *et al.* [111] investigated soaking times ranging from 0 to 84 hours. Optimal improvements were observed at a soaking time of 36 hours, and it was concluded that soaking time significantly influences the formation of secondary carbides. These findings were supported by SEM micrographs of cryogenically treated D2 steel at different soaking times, shown in Fig. 14. Oppenkowski *et al.* [109] reported that soaking

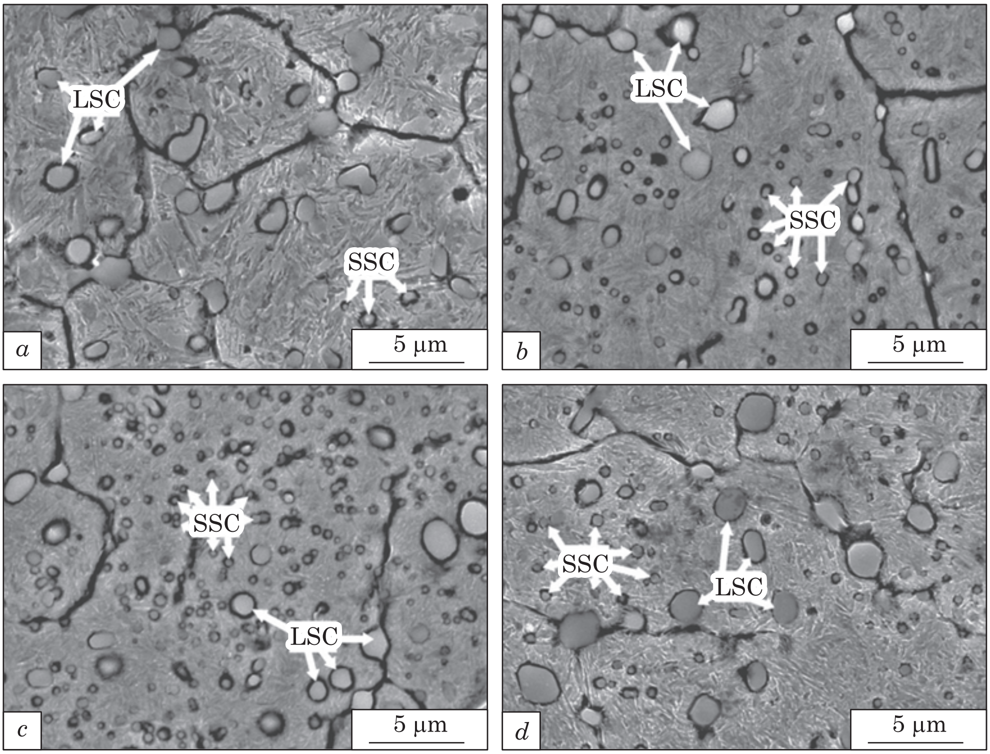


Fig. 14. SEM micrographs: conventional heat treatment (*a*), and cryogenic treatment at soaking times of (*b*) 12 h, (*c*) 36 h, (*d*) 84 h [111]

time has a significant effect on material properties. Maximum wear resistance was observed at a soaking time of 36 hours and decreased with further increases in soaking time. Collins *et al.* [110] reported a slight increase in hardness with longer soaking times, while the improvement in wear resistance was more pronounced with extended soaking periods.

In the studies [112, 113], finite element modelling of microstructure evolution during the combined drawing process with cryogenic cooling was carried out using the JMAK and cellular automata methods. Additionally, the simulation results were compared with laboratory experimental data. A comparison of the microstructure evolution obtained by both methods revealed a high degree of agreement across all considered models. After three deformation passes, an ultrafine-grained structure with an average grain size of 0.5 μm was obtained, consisting of a mixture of austenite and 88% α-martensite (Fig. 15). The level of martensite formation was found to be directly dependent on the intensity of metal cooling.

Among various methods of preparing martensitic materials with desired properties, *e.g.*, magnetic ones (see [114, 115] and references therein), there is the so-called 'strained-wire method', when a uniaxial tensile stress

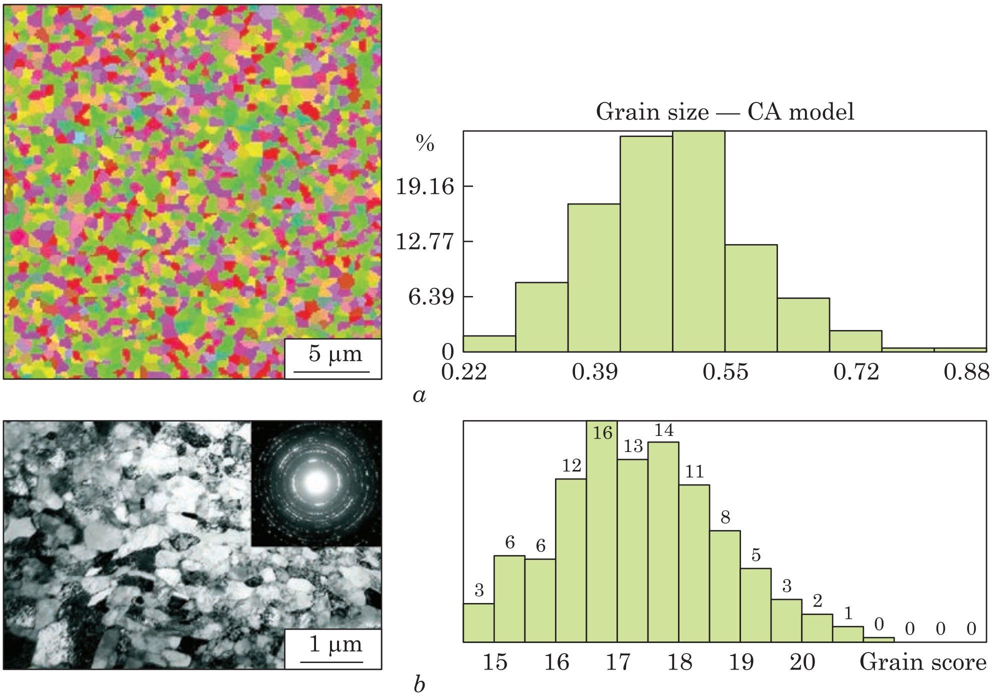


Fig. 15. Microstructure of AISI-316 steel after three deformation passes: (*a*) modelling [113]; (*b*) experiment [3]

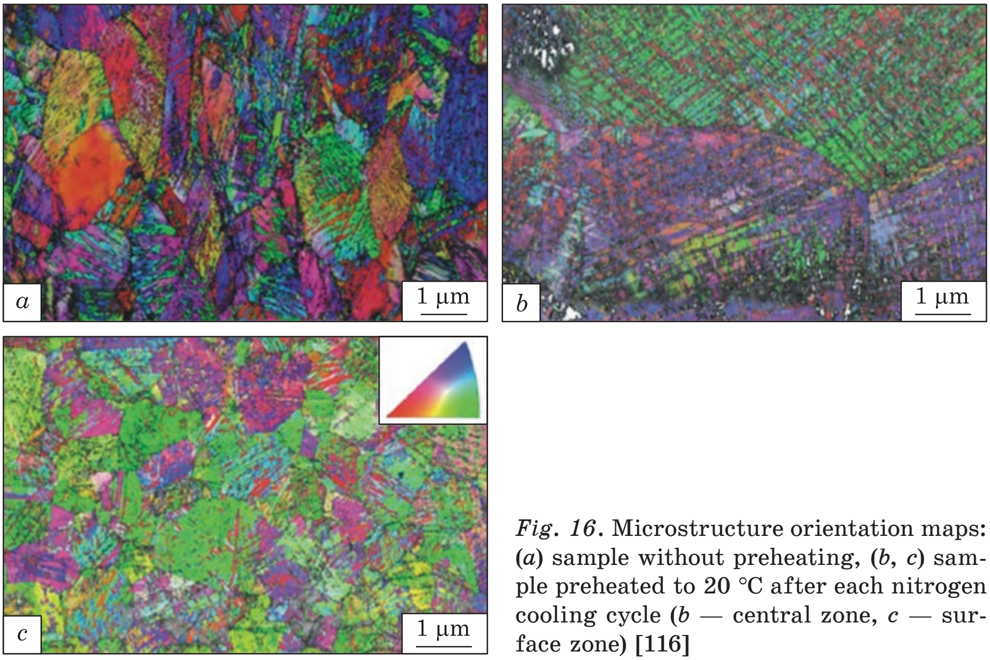


Fig. 16. Microstructure orientation maps: (*a*) sample without preheating, (*b*, *c*) sample preheated to 20 °C after each nitrogen cooling cycle (*b* — central zone, *c* — surface zone) [116]

is applied to the wire-shaped sample during the post-annealing stage. This method can be accompanied by CT as well.

In the wire subjected to deformation at cryogenic temperature without preheating, the observed microstructure consists predominantly of α′-martensite (98% of indexed points). The α′-martensite appears in banded structures. Using orientations obtained from residual austenite domains, considered as the initial orientations of the parent phase, the bands associated with α′-martensite form along the dense {111} deformation systems (Fig. 16, *a*). The grain size achieved by the α′-martensite is 0.5 μm [116].

In the wire subjected to deformation at cryogenic temperature with preheating, the amount of martensite differs between the central and surface zones. In the surface zone, the observed microstructure, similar to the wire deformed without preheating, consists predominantly of α′-martensite (98% of indexed points) (Fig. 16, *b*). In contrast, the microstructure at the centre shows markedly different results (Fig. 16, *c*). At this depth, austenite grains exhibit pronounced colour variations, reflecting intragranular misorientation caused by plastic deformation. In the orientation map, α′-martensite is represented with orientation colouring along the *y*-axis, normal to the processed surface, while several residual austenite domains are shown as contrasting bands. The observed microstructure consists of α′-martensite (68% of indexed points) and residual austenite. The morphology of the original austenite structure is poorly distinguishable, with multiple variants of α′-martensite present, as indicated by the diverse colours within the former austenite grains.

4. Conclusions

Cryogenic cooling has been used for many years as an environmentally friendly and efficiency-enhancing method; however, approximately 59% of studies in this field have been conducted since the 2000s. Despite the availability of commercial cryogenic processing equipment, this technique is currently not widely adopted in industry. This can be explained by the fact that, unlike conventional cutting fluids, cryogenic cooling does not allow the reuse of the coolant, and LN_2 evaporates even when not in use due to its high expansion properties. The high initial costs associated with transitioning to cryogenic cooling systems also limit the widespread implementation of this technology.

Although several studies report that cryogenic cooling affects the entire material, systematic investigations are required to verify experimentally its effectiveness across the full cross-section of the work piece. Many areas still lack quantitative studies. From the literature review presented above, it is clear that further research on the application of cryogenic cooling to both ferrous and non-ferrous metals could be conducted to enhance their mechanical properties and service performance.

Authors' Contributions. Z.G.M. reviewed and analysed the literature data for Sections 1 and 2: application of cryogenic cooling in machining processes. G.Z.S. reviewed and analysed the literature data for Sec. 3: the effect of cryogenic cooling on microstructural changes in metals. V.I.E. supervised the project, devised the main conceptual ideas, verified analytical approaches (all sections), and provided critical feedback. All authors approved the final version of the manuscript.

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ВПЛИВ КРІОГЕННОГО ОХОЛОДЖЕННЯ НА ЗМІНУ МІКРОСТРУКТУРИ МЕТАЛІВ

Дослідження кріогенного оброблення металів є предметом великої кількості наукових праць; тому написання оглядової статті для висвітлення сучасного стану знань і визначення напрямів майбутніх досліджень є вельми актуальним. Хоча вже є огляди щодо кріогенного оброблення інструментальних сталей, досі не було огляду, в якому б аналізувався вплив кріогенного оброблення на зміни мікроструктури металів. Тому детально розглянуто вплив окремих параметрів оброблення, їхня послідовність і вплив стабілізації за кімнатної температури на еволюцію мікроструктури. Оброблення матеріалів за кріогенної температури поліпшує такі їхні властивості, як твердість, міцність, зносостійкість, міцність на розрив, стабільність розмірів, корозійна стійкість тощо. Однак ступінь поліпшення властивостей матеріалів, підданих кріогенному обробленню, як зазначено в літературі, є різноманітним і іноді суперечливим. У межах виконаного дослідження зроблено спробу вичерпного аналізу численних наукових публікацій з цієї тематики.

Ключові слова: мікроструктура металів, оброблення металів, охолодження, кріогенне термооброблення.