

PAPER • OPEN ACCESS

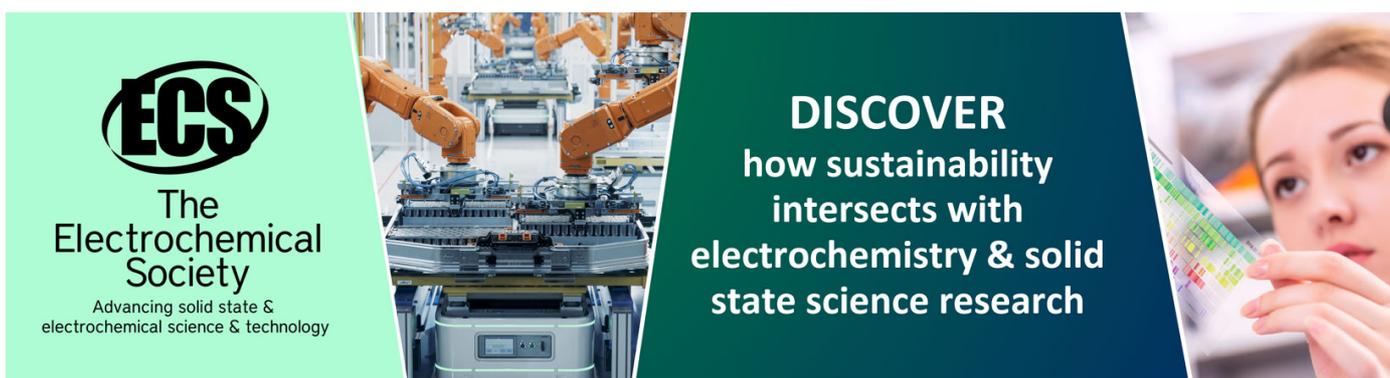
Resistance to brittle fracture and availability of austenitic steels

To cite this article: Boris Ermakov *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **450** 032041

View the [article online](#) for updates and enhancements.

You may also like

- [Analysis of research in the field of automatic production systems with rigid interaggregate relation and recomposed systems](#)
Sergej Bobrovskij, Boris Gorshkov, Semen Zaides *et al.*
- [Working position with recomposed production systems: devices](#)
Sergej Bobrovskij, Boris Gorshkov, Semen Zaides *et al.*
- [Organofluorine chemistry: promising growth areas and challenges](#)
Larisa V. Politanskaya, Galina A. Selivanova, Elena V. Panteleeva *et al.*



ECS
The
Electrochemical
Society
Advancing solid state &
electrochemical science & technology

DISCOVER
how sustainability
intersects with
electrochemistry & solid
state science research

Resistance to brittle fracture and availability of austenitic steels

Boris Ermakov¹, S Vologzhanina², Igor Bobrovskij^{3,4,6}, Nikolai Bobrovskij³ and Yaroslav Erisov⁵

¹ Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russian Federation

² Saint Petersburg National Research University of Information Technologies, Mechanics and Optics University ITMO, Saint Petersburg, Russian Federation

³ Togliatti State University, Togliatti, Russian Federation

⁴ Samara Scientific Center of the Russian Academy of Sciences, Samara, Russian Federation

⁵ Samara National Research University, Samara, Russian Federation

⁶ E-mail: bobri@yandex.ru

Abstract. The paper presents the results of studies on the availability and resistance to brittle fracture of perspective austenitic chromium-nickel-manganese cryogenic steels, depending on the concentration and ratio of the nitrogen and vanadium content. The optimum content of these elements in deformed steels determined, the results of the studies and recommendations on the doping system are confirmed by the results of full-scale tests of low-temperature equipment under internal pressure in liquid nitrogen.

1. Introduction

Energy independence is one of the most important tasks of each country. New production technologies are being developed, including additive technologies [1-4]. New technologies for machining and post-processing appear [5, 6]. New methods of instrumental control tools for machining steels and alloys appear [7, 8]. Much attention is paid to environmental issues of steel products production [9, 10]. Sharp increase of energy consumption leads to increasing consumption rate of non-renewable oil reserve, which may have been already exhausted in our century.

The task of transition to alternative more affordable and cheaper fuels should be solved for the further development of transport, industry, provision of vital activity of the population in the coming decades. First of all, the task of transition to alternative fuels is the most actual in aviation, which currently consumes up to 25-30% of annual fuel consumption, and this segment is projected to grow by several percent per year [11].

Considering the geographic features, Russian Federation needs for accelerated creation of alternative fuels. Cryogenic fuels based on liquid hydrogen are the most perspective of them. Cryogenic fuel is able to compete on energy content with oil-based fuels and it is more environmentally friendly, practically not polluting the environment.

However, the introduction of cryogenic fuels gives a number of new challenges. The most important task: development of new economically-alloyed cryogenic steels.



2. Theoretical analysis and statement of problems

There are a number of parts and assemblies in cryogenic technology, to the metal of which there are no requirements for the stability of the austenitic structure during operation [11-13]. These include tanks for storage and transportation of liquefied gases, fuel tanks of cars operating on liquefied natural gas, as well as fuel tanks for aerospace equipment. High specific strength and manufacturability are the main metal's characteristics for manufacture. However, traditionally such tanks are made of 12X18H10T steel, its specific strength is low, which for a long time restrained the widespread use of this type of fuel. The purpose of this work was to confirm the possibility of using Cr-Ni-Mn metastable austenitic steels, additionally alloyed with vanadium and nitrogen as materials for parts, assemblies and structures of cryogenic fuel equipment. Our studies showed that equipment made from these steels has the necessary strength reserves in combination with a high complex of plastic and viscous properties at low temperatures.

Chromium-nickel-manganese metastable austenitic steels have an unusual complex of mechanical properties. The relatively low yield strength is combined with high values of point of maximum load at room temperatures, sufficient ductility and fracture toughness at cryogenic temperatures. These features are caused by the deformation martensitic transformations (DMF) during the plastic deformation and the high ability to deformation hardening of austenite caused by these transformations [12, 13, 16]. High values of plastic characteristics and impact toughness at cryogenic temperatures caused by the martensitic $\gamma \rightarrow \alpha$ - transformation occurring at low-temperature deformation and also the effect of "ductility induced by the transformation" (PNP effect) associated with it.

The average rate of formation of α -martensite under proportional deformation by static stretching at given temperature conditions is the criterion for the intensity of phase transformations [13]:

$$M = \alpha / \delta \quad (1)$$

It is the ratio of the α - amount of martensite formed in the zone of proportional deformation of the sample at a given temperature to δ - relative elongation at the same temperature. Such a criterion most fully describes the relationship between the kinetics of phase transformations and the mechanical properties of metastable austenitic steels.

3. Materials and experimental technique

Metastable austenitic steels (MAS) have been known for a long time, but have not yet found wide application in cryogenic technology, although for a number of products and structures, which are not required to maintain the stability of magnetic properties, the use of these steels is very promising. Comparative tests of low-temperature mechanical properties of steels and alloys widely used in cryogenic engineering and new metastable austenitic steels additionally doped with nitrogen and vanadium were carried out in this work. The melting of the experimental steels was carried out in an open high-frequency induction furnace with a basic lining under the flux of 92% CaF₂, 3.5% CaO, 2% SiO₂, 1.8% Al₂O₃, and a number of micro additives.

During melting, a specially prepared marten-batch charge was used as the matrix substrate with following chemical composition (mass, %): carbon-0.03; chromium-0.25; manganese - 0.67; aluminum - 0,03; nickel - 0.20; sulfur and phosphorus – less than 0.01 each. Carbon-free ferrochromium, metallic manganese, ferrovandium, electrolytic nickel, manganese with a nitrogen content of 5.9%, or ferrochrome containing 7.4% nitrogen were used as alloying materials. During nitrogen-containing melts, nitrogen doping was performed at a temperature of 1750-1770 K, after the introduction of nitrogen, the temperature of the melt was maintained in the range of 1740-1780 K. Before the introduction of the nitrided materials, the melt was deoxidized by aluminum ingots at a rate of 1 g per 1 kg of melt and SF-45 lump ferrosilicon. Finally, the deoxidation was made with calcium metal. Optimization of experimental steels' compositions was carried out by the method of mathematical planning of the experiment. The mechanical properties and operability of experimental steels at cryogenic temperatures were investigated in the temperature range 293-20 K using standard techniques [14, 15]. Thus, uniaxial static tension tests were carried out according to GOST 1497-84 on type I

samples. For tests at low temperatures, a type I cryostat [11] was used - a cryostat with two-way entry of power elements. Liquid nitrogen - 77 K and helium vapor was used as cooling medium for testing at a temperature of 20 K. Dynamic tests were carried out on specialized copra with the placement of a loading system inside the refrigerating chamber. Uniaxial static tension testing was carried out in accordance with GOST 1497 -84 on samples of type I. Tests of containers under internal pressure were carried out by the method of NGO Cryogenmash [11].

4. Metastable austenitic steels for cryogenic technology

Figure 1 shows the dependence of strength and toughness on specimens with an acute incision on the degree of austenite instability at 293, 77, and 20 K. According to the given data, the maximum value of the toughness is achieved for steels with a criterion M equal to approximately 1.2 and 1.8 at temperatures of 77 and 20 K, respectively.

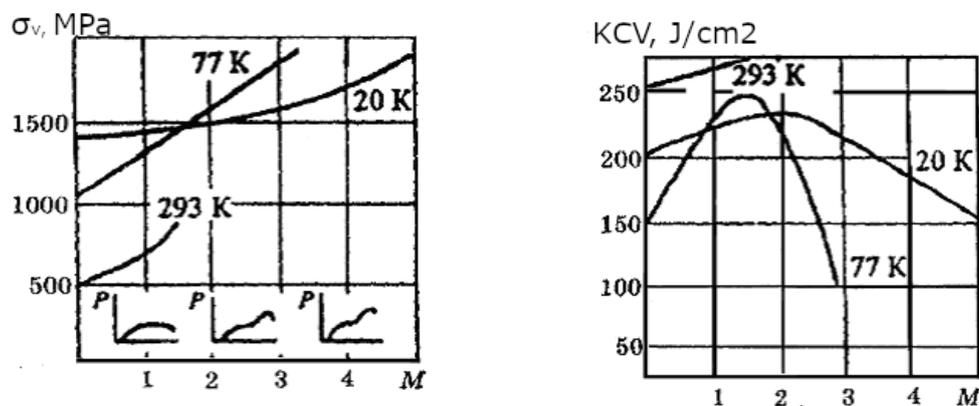


Figure 1. Dependence of the point of maximum load (left) and toughness (right) on the degree of instability of austenite in the MAS of type 06X15N9G8AF with different ratio $[V] / [N+C]$.

This is correlates with the fact that at $M < 1.277$ K and $M < 1.8$ at 20 K the neck does not have time to harden to such an extent that it ceases to be the weakest point of the sample's working part, and at $M > 1.2$ at 77 K and $M > 1.8$ at 20 K, the martensitic transformation intensifies, which leads to premature destruction of the sample by a martensitic structure with a smaller margin of plasticity. The condition $[V] / [NC] = 3.1$ corresponds to such values of the criterion M in the Cr-Ni-Mn MAS, when the most complete binding of nitrogen, carbon and vanadium to carbonitride V (CN) occurs.

In the process of aging of Cr-Ni-Mn MAS additionally doped with vanadium and nitrogen, two opposite processes occur simultaneously: softening of the solid solution as a result of removal of the strengthening element, nitrogen, from the solid solution, while increasing the strength of the material by separating the carbonitride phases. However, the strengthening effect of carbonitrides is significantly higher than the softening effect of nitrogen excretion. As a result, the strength properties of steels, especially their yield stress, increase sharply. Fig. 2 shows the influence of the ratio $[V] / [C+N]$ on the mechanical properties of metastable austenitic steels based on Cr-Ni-Mn with nitrogen and vanadium.

Table 1 shows the optimal composition of Cr-Ni-Mn MAS with nitrogen and vanadium taking into account the martensitic transformation criterion.

Table 1. The chemical composition of steel 06X15N9G8AF.

C	Cr	Ni	Mn	V	N	S	P
≤0.06	14 - 16	8.5 - 9.5	7 - 9	1.0 - 1.5	0.2-0.4	≤0.025	≤0.025

Heat treatment of steel 06H15N9G8AF consists of hot rolling the sheet at a temperature of 1500-1200 K, austenitization at 1425 K with oil cooling. The mechanical properties of steel are given in table 2.

Table 2. Mechanical properties of metastable austenitic steel 06X15N9G8AF.

T (K)	σ_B (MPa)	$\sigma_{0.2}$ (MPa)	δ_5 (%)	Ψ (%)	KCV (J/cm ²)
293	725	375	61	77	275
100	1120	540	60	75	240
77	1600	800	58	75	210

Table 3 presents the comparison of the material’s mechanical properties used for the manufacture of pressure containers in cryogenic technology.

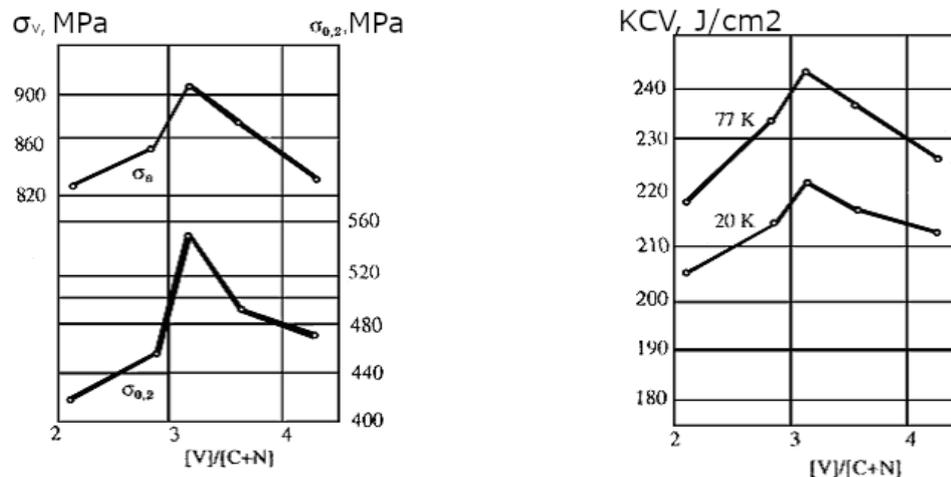


Figure 2. Dependence of strength (left) and toughness (right) of MAS of the type 06X15N9G8AF with nitrogen and vanadium on the ratio [V] / [C+N] after austenitization at 1425 K with oil cooling and subsequent aging at 980 K for 25 hours.

Data analysis of the Table 3 allows us to recommend the usage of metastable austenitic steel 06X15N9G8AF along with titanium alloys for cryogenic equipment. However, titanium is characterized by low hardness, its modulus of elasticity is half of the modulus of elasticity of metastable steel. In addition, titanium alloys are not suitable for impact loads in contact with liquid oxygen and other less common fluorine-based oxidants, which narrows the scope of its possible use in liquid rocket engines.

Table 3. Comparative characteristics of materials used for the manufacture of pressure containers in cryogenic technology.

Material	σ_v	$\sigma_{0.2}$	δ_5	KCV		Elasticity modulus E (MPa•10 ⁴)	High specific strength $\sigma_{0.2}$ /(γ,g) (km)	
	(MPa)	(MPa)	(%)	(J/cm ²)				
	Temperature (K)							
				293	77	20		
AMg5	280	127	23	15			6.9	47
BT1-Oct	470	400	48	24			10.7	89
12X18N10T	529	235	37	34	280	250	18.6	30
06X15N9G8	650	260	50	42	247	224	20	33
06X15N9G8AF	900	500	46	36	240	221	20	64

For a comprehensive study of the steel 06X15N9G8AF properties, the containers were tested for bursting with an internal pressure of liquid nitrogen (Table 4).

Table 4. The results of the container's test by internal pressure; work medium - liquid nitrogen.

Steel	σ_v	$\sigma_{0.2}$	δ_5		KCV		Burst pressure (MPa)	Stresses in the wall at the time of destruction (MPa)	Destruction place
	(MPa)	(MPa)	(%)	(%)	(J/cm ²)	(J/cm ²)			
	Temperature (K)								
	293	293	77	20	77	20			
12X18N10T	660	260	37	32	300	280	9.8-13.2	490-660	Zone of thermal influence of longitudinal seam
06X15N9G8AF	900	500	46	36	242	220	18.0-21.0	1038-1240	Girth seam

The positive results obtained during the tests and the proven technology of production and processing allowed us to recommend the steel 06X15N9G8AF as a material for fuel tanks of cryogenic designation. At present, the issue of the production of fuel tanks for liquid hydrogen from this steel is being considered.

5. Conclusions

The results of the study confirmed the availability of metastable austenitic steels usage as materials for cryogenic equipment. The properties of MAS in deformed states correspond to the requirements imposed on fuel cryogenic systems. The obtained data on the MAS efficiency made it possible to draw the following conclusions:

1. The availability of MAS usage in deformed state as materials for elements of fuel equipment for cryogenic purposes is presented.
2. Doping with vanadium dramatically increases the entire range of performance properties of austenitic steels, including their resistance to brittle fracture during prolonged low-temperature operation. At present, a number of austenitic vanadium-containing steels possessing an increased level of resistance to brittle fracture during a long service life at negative temperatures have been developed, tested and recommended for use as materials of low-temperature and cryogenic technology. These steels include: steel 06X15N9G8AF, from which the experimental industrial lot of fuel cryogenic containers was manufactured and steel 07X13G28ANFL, which is recommended for a number of elements of locking and regulating equipment.

References

- [1] Smelov V G, Sotov A V and Agapovichev A V 2016 Study of structures and mechanical properties of products manufactured via selective laser sintering of 316L steel powder *Chernye Metally* **9** 61-5
- [2] Alexeev V P, Balyakin A V, Khaimovich A I 2017 *IOP Conf. Ser.: Mater. Sci. Eng.* **177(1)** 012120
- [3] Grigoriev S N, Bobrovskij N M, Bobrovskij I N and Jiang C –P 2017 Technological parameters forming the surface texture in hyper productive surface Plastic deformation processing *Key Engineering Materials* **746 KEM** pp 114-9
- [4] Bobrovskij N M, Melnikov P A, Grigoriev S N and Bobrovskij I N 2015 *IOP Conf. Ser.: Mater. Sci. Eng.* **91(1)** 012034
- [5] Bobrovskij I N 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **302** 012041
- [6] Bobrovskij I N 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **302** 012066
- [7] Lukyanov A A, Grigoriev S N, Bobrovskij I N, Melnikov P A and Bobrovskij N M 2017 *IOP Conf. Ser.: Earth Environ. Sci.* **66** 012020
- [8] Bobrovskij N M, Melnikov P A, Grigoriev S N and Bobrovskij I N 2015 *IOP Conf. Ser.: Mater.*

- Sci. Eng.* **91(1)** 012035
- [9] Grigoriev S N, Bobrovskij N M, Bobrovskij I N, Melnikov P A and Lukyanov A A 2017 *IOP Conf. Ser.: Mater. Sci. Eng.* **50** 012015
- [10] Grigoriev S N, Bobrovskij N M, Melnikov P A, Bobrovskij I N and Zaborowski T 2017 Research of Tool Durability in Surface Plastic Deformation by Wide Burnishing of Cast Iron without Metalworking Fluids *Key Engineering Materials* **746** 120-5
- [11] Solncev Yu P and Stepanov G A 1982 *Materials in creogenic technology* (Leningrad: Mashinostroenie) p 312
- [12] Solncev Yu P, Ermakov B S and Povarov D V 2007 *Materials and reliability of low-temperature structures* (SPb: HIMIZDAT) p 168
- [13] Solncev Yu P and Ermakov B S 2006 *Resource of materials with low-temperature structures* (SPb: HIMIZDAT) p 512
- [14] Brodskij V Z, Brodskij L I and Golikova T I 1982 *Table of experiment plans for factor and polynomial models*
- [15] Fridman Ya B 1974 *Mechanical properties of metals* (Moscow: Mashinostroenie) p 368
- [16] Grikurov G N 1994 *FMM* **78(1)** 114-21
- [17] Solncev Yu P, Andreev A K and Grechin R I 1989 Casting cold-resistant steel (Moscow: Metallurgiya) p 176