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### INFLUENCE OF TEMPERATURE OF THERMAL PROCESSING ON INTERCRYSTALLINE CORROSION RESISTANCE OF WELDED JOINTS

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#### Abstract

Welded joints with corrosion-resistant steels and heat-resistant alloys requiring different modes of heat treatment to achieve the level of mechanical properties specified in the design documentation are used to manufacture parts and components of the rocket engine turbo-pump unit.

For the manufacture of parts and assemblies of liquid-propellant rocket engines at the machine-building enterprises of Ukraine there was a necessity of replacement of half-finished products because of certain difficulties at delivery of materials from EU countries.

First of all, it was necessary to replace flat products from high-alloy ХН67МВТЮ and 06Х15Н6МВФБ with one alloy that would possess the necessary set of physicomechanical characteristics. In the work, as a replacement for the used heat-resistant alloys, the Inconel 718 alloy welded to 316L steel was chosen. As a result of comparative studies of the intercrystalline corrosion resistance of welded joints of the heat-resistant Inconel 718 alloy with stainless steel, after testing various heat treatment conditions, the low-temperature heating conditions were recommended for soldering at a temperature of 950 °C. Samples of welded joints, processed according to the recommended mode, showed increased corrosion resistance.

**Keywords:** heat-resistant alloy; corrosion resistance; intercrystalline corrosion; welding; structure; soldering.

### ВПЛИВ ТЕМПЕРАТУРИ ТЕРМІЧНОЇ ОБРОБКИ НА СТІЙКІСТЬ ДО МІЖКРИСТАЛІТНОЇ КОРОЗІЇ ЗВАРНИХ З'ЄДНАНЬ

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#### Анотація

Для виготовлення деталей і вузлів турбонасосного агрегату ракетного двигуна застосовуються зварні з'єднання з корозійностійкими сталями і жароміцними сплавами, які вимагають різних режимів термічної обробки для досягнення рівня механічних властивостей, зазначених в конструкторській документації.

При виготовленні деталей і вузлів рідинних ракетних двигунів, у зв'язку з певними труднощами при поставчанні матеріалів з країн ЄС, на машинобудівних підприємствах України виникла необхідність заміни напівфабрикатів. В першу чергу потрібна заміна листового прокату із високолегованих сплавів ХН67МВТЮ і 06Х15Н6МВФБ на один сплав, який володів би необхідним комплексом фізико-механічних характеристик. У роботі в якості заміни застосовуваних жароміцних сплавів обраний сплав Inconel 718 у зварному з'єднанні зі сталлю 316L. У результаті порівняльних досліджень стійкості до міжкристалітної корозії зварних з'єднань жароміцного сплаву Inconel 718 нержавіючої після різних режимів термічної обробки рекомендований режим низькотемпературного нагріву при пайці при температурі 950 °C. Зразки зварних з'єднань, оброблені за рекомендованим режимом, показали підвищену корозійну стійкість.

**Ключові слова:** жароміцний сплав; корозійна стійкість; міжкристалітна корозія; зварювання; структура, пайка.

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## ВЛИЯНИЕ ТЕМПЕРАТУРЫ ТЕРМИЧЕСКОЙ ОБРАБОТКИ НА СТОЙКОСТЬ К МЕЖКРИСТАЛЛИТНОЙ КОРРОЗИИ СВАРНЫХ СОЕДИНЕНИЙ

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### Аннотация

Для изготовления деталей и узлов турбонасосного агрегата ракетного двигателя применяются сварные соединения с коррозионностойкими сталями и жаропрочными сплавами, требующие различных режимов термической обработки для достижения уровня механических свойств, указанных в конструкторской документации.

При изготовлении деталей и узлов жидкостных ракетных двигателей, в связи с определенными трудностями при поставке материалов из стран ЕС, на машиностроительных предприятиях Украины возникла необходимость замены полуфабрикатов. В первую очередь необходима замена листового проката из высоколегированных сплавов ХН67МВТЮ и 06Х15Н6МВФБ на один сплав с высоким комплексом физико-механических характеристик. В работе в качестве замены применяемых жаропрочных сплавов выбран сплав Inconel 718 в сварном соединении со сталью 316L. В результате сравнительных исследований стойкости к межкристаллитной коррозии сварных соединений жаропрочного сплава Inconel 718 с нержавеющей сталью после различных режимов термической обработки рекомендован режим низкотемпературного нагрева при пайке при температуре 950 °С. Образцы сварных соединений, обработанные по рекомендованному режиму, показали повышенную коррозионную стойкость.

**Ключевые слова:** жаропрочный сплав; коррозионная стойкость; межкристаллитная коррозия; сварка; структура; пайка.

### Introduction

The main operational properties of turbo-pump engine parts and units (TPA) are heat resistance, long-term durability and corrosion resistance [1–3]. These requirements are achieved by selection of materials, technology of their production and subsequent processing. Thermal influences during welding and heat treatment affect the alloy structure and change performance properties. Therefore, the effect of thermal influences on the corrosion resistance of welded joints of the main combinations of materials used in the missile carrier manufacture was studied during the work. Since most parts and assemblies of TPA are soldered and welded in different sequences, the soldering of heat-resistant alloy using band solder after corrosion-resistant steel welding was considered. This work was carried out to study various thermal influences of the soldering process on a welded joint to simulate the conditions for manufacturing real assemblies.

### Problem Overview

Corrosion is a spontaneous redox process of destruction of metals and alloys due to interaction with the environment. When multi-component alloys come into contact with an electrolyte (which can be water adsorbed from air), many micro-galvanic pairs appear on the metal surface. In these pairs, atoms of a more active metal (usually Fe) play the role of an *anode*, and atoms of a less active metal play the role of a *cathode*. At the cathode, there is a reduction process of O<sub>2</sub> molecules in neutral and alkaline me-

dioms, or of H<sup>+</sup> ions in an acidic medium [5; 6; 8; 9].

Iron oxides Fe<sub>2</sub>O<sub>3</sub> form a loose film does not prevent the penetration of air oxygen and corrosion. Corrosion resistance of steels is achieved by alloying steels with chromium or chromium and nickel [4]. Corrosion-resistant steels can be classified by structural class as ferritic, pearlitic, martensitic and austenitic – depending on the structure after heating the steels in the austenite field and cooling in air [9–13].

Nickel is the basis of the Inconel 718 alloy; it has an FCC crystal lattice similar to the Fcc lattice. Therefore, nickel doping broadens the austenite field [27] (Fig. 1).

The most widely used steels are nickel-chromium steels of 12X18H9 and 12X18H9T grades. The structure of these steels is austenitic, both at room temperature and upon heating, i.e. they do not undergo polymorphic transformation. Products made of these steels are subjected to heat treatment, but not for the purpose of hardening (this is impossible, since recrystallization does not occur with a change in temperature), but to increase corrosion resistance due to the chromium enrichment of solid solution [14–17]. Presence of carbon means that chromium carbides are present in the structure of the annealed steel, which means that not all chromium is in solid solution. Quench hardening at a ~1000°C temperature allows to dissolve the chromium carbides in austenite and to prevent their precipitation during quenching.

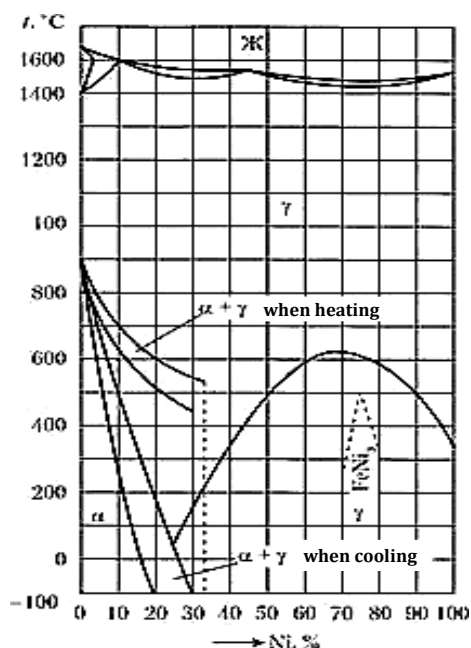


Fig. 1. System Status Chart "Fe - Ni" [27]

## Materials and Methods

The Inconel 718 heat-resistant austenitic alloy of the Fe-Cr-Ni system and the 12X18H10T and SLS 316L corrosion-resistant steels were selected as the materials for the study. Inconel 718 alloy is a heat-resistant nickel alloy of the Fe-Cr-Ni system and is used in rocket engineering for the manufacture of rocket engine combustion chamber jackets, parts of turbopump assemblies and other critical duty products [18–22]. The alloy is used in the temperature range from  $-252.8\text{ }^{\circ}\text{C}$  to  $+704.4\text{ }^{\circ}\text{C}$ , and is stable in chemically active gaseous environments at the temperature of up to  $+980\text{ }^{\circ}\text{C}$  [23–26].

316L steel is austenitic structural cryogenic steel. This steel is resistant to corrosion in aggressive environments, as well as to most external influences, it has the ability to maintain the structure integrity during an increase or decrease of temperature [26; 28]. The studied samples of 316L steel were obtained using powder raw materials and additive technology [29].

The studied materials and chemical composition are given in Table 1.

Table 1

The chemical composition of the investigated alloys

Alloy	Semi-fabricated product, mm	Chemical composition, %													
		C	Mn	Si	S	P	Cr	Ni	Ti	Mo	W	V	Al	Cu	Fe
Inconel 718	Sheet 3.17	0.03	0.08	0.08	0.0001	0.008	18.24	53.53	0.97	2.99	-	B-0.002	0.51	0.07	17.9
	Filler wire $\varnothing$ 1.6	0.04	0.06	0.07	0.001	0.008	18.75	53.44	0.98	2.88	-	B-0.004	0.58	0.11	17.8
12X18H10T	Sheet 3.0	0.05	0.57	0.59	0.002	0.029	17.58	9.05	0.3	0.1	0.023	0.029	-	0.22	Basis
316L	Sheet 3.0	0.03	2.0	1.0	0.003	0.045	16.0	14.05	0.5	3.1	0.023	-	-	-	Basis

Tests and studies were performed on the equipment of a machine-building enterprise. The ICC tests were carried out using the "AM" method in accordance with GOST 6032-89 [30]: the welded samples were boiled for 24 hours in a sulfuric acid solution with the addition of copper sulfate and copper chips, then the samples were bent by 90 degrees. The bending point was controlled by visual inspection and metallographic method using MBC and Neophot-2 microscopes with magnification up to 200 times.  $5 \times 10$  mm samples for the manufacture of metallographic specimens

were cut from the bending sections. Heat treatment was carried out in a СП-1300 heat-treatment silit furnace, Г70HX soldering was simulated in accordance with soldering conditions and a standard process, welding was performed with a CAHE-2M welding machine.

## Discussion

*Weld preparation.* Before welding, each workpiece underwent geometry control, magnet check and visual inspection for the presence of oxide scabs and nicks. In accordance with OST 92-1152-75, before welding, each weld edge was

cleaned of oxide films formed during heat treatment and degreased with a petroleum solvent.

**Welding process.** Welded assemblies were welded by manual argon-arc welding in accordance with the recommendations of the Nicrofer manufacturer: I = 140 A, argon consumption: 15 l/min for protection, 5 l/min for blowing. During shielded arc welding, grade 1 argon was used (volume fraction of argon – 99.90%, oxygen – 0.005%, nitrogen – 0.10 %, water vapor at 760 mm Hg – 0.03 g/m<sup>3</sup>).

**Weld joint treatment.** To remove the oxide film and oxidation tint, the surface of the weld joint and the weld-affected zone was treated to a Rz 40 roughness parameter and all welded joints underwent X-ray control for welding defects. According to the manufacturing process, after welding workpieces were sent for Г70HX band soldering (the solder contains Mg 70 %, as well as additional components – nickel, chromium).

Soldering was carried out in two modes:

– heating to 950±10 °C, holding for 30 minutes from the moment of loading into the furnace, cooling to 300 °C in the furnace, then in the air.

– heating to 1200±10 °C, holding for 20 minutes from the moment of loading into the furnace, cooling to 300 °C in the furnace, then in the air.

**Determination of ICC resistance.** To determine the ICC susceptibility, either welded samples or

samples after special ICC provoking heat treatment are tested. The provocative treatment conditions depend on the structural class of steel. Standard test methods and standard steels are specified in GOST 6032-89. For new non-standardized steels, the MCC control method is selected taking into account the environment for which the steel is intended.

The study of welded samples of Inconel 718-stainless steel alloy for ICC resistance was carried out on the steel side. The 316L steel surface in the weld zone was examined in the as-received condition (condition of supply), without surface conditioning. The results of the study are given in Table 2.

Etching treatment was carried out using a reagent of the composition: water – 1000 cm<sup>3</sup>, copper sulfate – 130.0 g, sulfuric acid – 120.0 g. In accordance with the GOST requirements, the sample is considered resistant if the ICC depth does not exceed 0.03 mm. Increased etchability is not considered a defect, but it indicates a tendency for the material to corrode (Table 2). When identifying ICC using structural etching, it should be noted that grain boundaries are thermodynamically more active and etched more strongly than the rest of the grain surface. Therefore, etched grain boundaries can be mistaken for ICC.

Table 2

The results of research on intercrystalline corrosion				
Sample No.	Material	Heat treatment conditions	ICC depth, mm	Conclusion
132	STS 316L - inconel 718	Soldering imitation 950 °C+ aging	No ICC (increased etchability to a 0.33 mm depth)	Resistant
133		Soldering imitation 1200 °C+ aging		
151	12X18H10T- inconel 718	After soldering:	0.24	Not resistant
141		Soldering imitation 1210 °C+ aging	0.17, (increased etchability to a 0.23 mm depth)	
2		Soldering imitation 950 °C+ aging	0.07, (increased etchability to a 0.14 mm depth)	

Before ICC susceptibility testing, a visual inspection and a metallographic study (with a welded sample surface magnification up to 200 times) on the 316L steel side were performed. It was established that the surface of the samples from the initial side is rough with the presence of recesses and protrusions of oblong and round shape, the other side is smooth and slightly rough after mechanical cleaning.

During microexamination of sections of the samples No. 132 and 133 intercrystalline corrosion was not detected. From the surface to a depth of 0.33 mm, increased etchability of grain boundaries was observed due to the precipitation of various oxide inclusions. During visual inspection, recesses up to ~0.08 mm were found (Fig. 2) in sections on the initial surface side of samples No. 132 (Fig. 2a) and No. 133 (Fig. 2b).

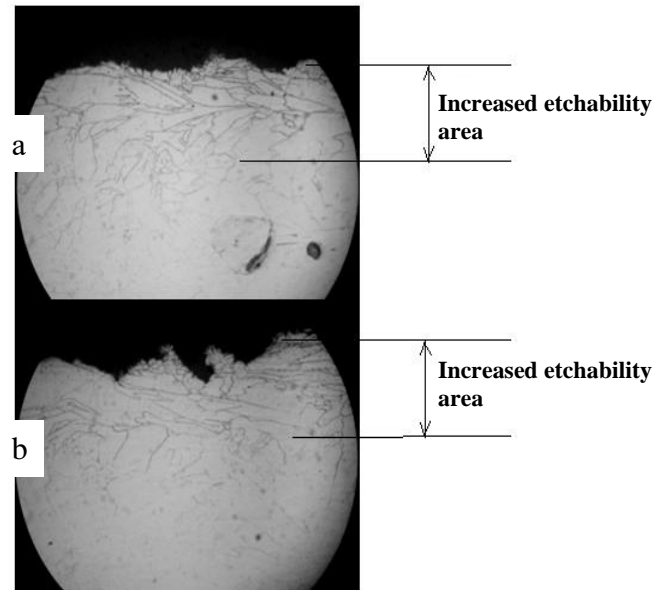


Fig. 2. Increased etchability of grains from the sample surface from the side of steel STS 316L, x200

Increased etchability of the grain boundaries of 316L steel indicates the ICC susceptibility of the alloy, but it is not definitive. When increased etchability of grain boundaries is detected in the steel structure study, it is important to note its depth. Increased etchability of grain boundaries near the edge of the sample also occurs during electrolytic etching due to the higher current density at the edge.

When examining samples that were soldered at a 1200 °C temperature, it was found that they are less resistant to ICC than those that have been soldered at a 950 °C temperature. Intercrystalline corrosion on 12X18H10T steel samples is shown in Fig. 3. The occurrence of susceptibility to corrosion damage of boundaries when steel is exposed to a corrosive environment is obviously associated with the occurrence of new grain boundary phases or segregations that differ in

composition from the average chemical composition of steel.

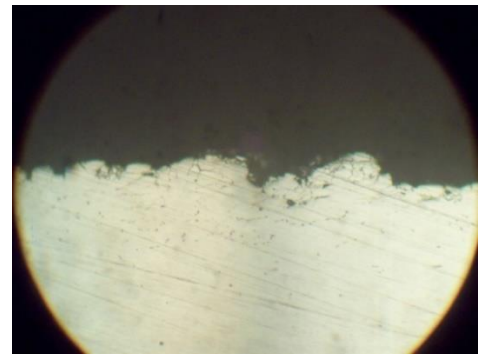


Fig. 3. Intergranular corrosion on steel samples 12X18H10T, x200

Grain corrosion areas are clearly visible in Fig. 3, which indicates that 12X18H10T steel is not ICC-resistant after soldering at a 1200 °C temperature.

The etching pattern of the steel grain boundaries during the ICC testing is shown in Fig. 4.

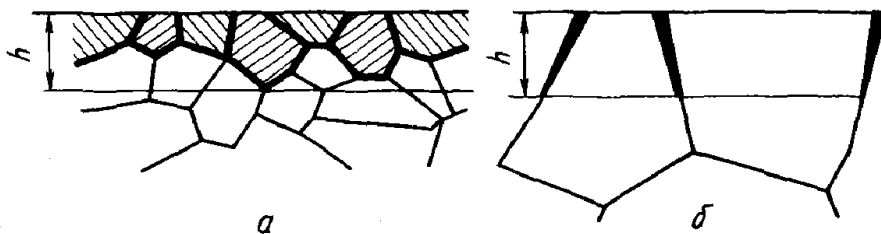


Fig. 4 Etching microstructure of steels:  
a - ICC; b - grain boundary etchability

ICC susceptibility occurs as a result of structural heterogeneity of grain boundaries, secondary phase precipitation, depletion or enrichment of adjacent sections of the  $\alpha$ -solid matrix solution of alloying elements, formation of submicro- and

micro-cavities through the drainage and coagulation of vacancies.

### Conclusions

1. A comparative study of the ICC susceptibility of Inconel 718-stainless steel alloy welded

joints at various soldering temperatures was carried out.

2. The study of welded samples of heat-resistant alloy with stainless steel for ICC resistance showed that it is not advisable to use welded joints of Inconel 718 alloy with 12X18H10T steel and 316L steel when soldering in aggressive environments at a 1200°C temperature.

3. Recommended heat treatment conditions: heating to  $950 \pm 10$  °C, holding for 30 minutes, cooling to 300 °C in the furnace, then in the air.

Welded samples of Inconel 718 alloy with 316L steel, treated in accordance with the recommended heat treatment conditions, showed high ICC resistance.

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