DEVELOPMENT OF ROLLING MODES FOR SAMPLES MADE FROM NICHROME POWDER ALLOY AND THEIR TESTING AT OPERATING TEMPERATURES

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Abstract *The rolling modes for samples of various thicknesses made of dispersed-hardened powder nichrome alloy have been developed. An alloy sample has been tested at operating temperatures on a stand of the Institute of Strength Problems of the National Academy of Sciences of Ukraine. It has been demonstrated that the existing set of properties makes it possible to use the created alloy for aerospace aircraft, which repeatedly operate at extremely high temperatures under aerodynamic heating conditions.*
***KEYWORDS**: DISPERSED-HARDENED POWDER NICHROME ALLOY, HIGH TEMERATURES, ROLLING*

1. Introduction

For thermal protection of reusable space systems of Shuttle and Buran type light carbon-carbon materials and superlight tale heat protection made of lightweight fibrous on the basis of quartz filaments with a specific weight of 110-250 kg/m^3 heated up to 1250 °C had been used [1-3]. However, such thermal protection has relatively low mechanical and thermo erosion characteristics. Furthermore such thermal protection had often been destroyed due to influence of solid particles and accidental impacts. For example, mechanical damage of carbon-carbon coatings at reusable space craft "Columbia" results to its catastrophe. That is why new ceramic and metallic materials with good mechanical strength which are more suitable for repair at ground service are now used for design of new kinds of thermal protection systems. On the base of the investigations of nonequilibrium processes in self organizing systems [4] dispersed hardened nichrome alloy [6, 7] for thermal protection systems had been proposed in [5].

However some problems had been appeared during the sample production. During the production of the samples of required sizes by rolling method the cracks in massive samples had been detected. It is necessary to use the special modes of treatment for production of the samples of required thickness.

The main aim of this work is the development of the modes of rolling for the samples of dispersed hardened alloys on the base of nichrome with the following their testing at the service temperatures up to 1200 ^{o}C at the special stand of Pisarenko Institute for Problems of Strength of National Academy of Sciences of Ukraine [8-10].

2. The aim of investigation

It is known that simultaneous adding of chromium and aluminum to nickel based alloys results to reduction of their density and increasing of their heat resistance and long term heat resistance. However at the same time their level of plasticity reduced due to formation of reinforcing phase $\gamma'(Ni_3Al)$ [11].

It is impossible to produce the samples with required density by means of increasing of pressing pressure that is why for increasing of density it is necessary to introduce the operations of additional pressing or rolling. Using of additional pressing for big samples is not reasonable because of press form wear enhancing. So the using of rolling is preferable.

The first attempts of rolling for new heat resistant nanohardened alloy based on Ni-Cr at the temperature of porous billets of 1120-1150 ^{o}C were unsuccessful. The attempts to change the rolling speeds, rollers diameter and thicknesses were unsuccessful too. Directed testing of all these processes required essential increasing of the number of billets to develop and confirm the rolling modes for such kind of alloys.

During the development of the production technologies for the sheets, tapes and foils from the new heat resistant alloys on the base of nichrome it was established empirically that such material has the recrystallization temperature of 0,9T_m (about 1200 ^{0}C). That is why the rolling at the billet temperatures up to 1120-1150 ^{0}C cannot be successful.

It was established that developed heat resistant materials characterized by sufficient plasticity in porous state even at room temperatures is catastrophically hardened during rolling and if compression ratio exceeds 10% its hardness exceeds 40HRC. All these factors required the special long term efforts for development of rolling mode for such kinds of alloys.

3. Rolling of billets from dispersed- hardened nichrome based alloy

The presence of open porosity does not allow heating the materials before and during rolling at air environment. During the heating till the temperatures more than 1000 ^{o}C the alloy structure is saturated by oxygen and becomes brittle. To have a possibility to make the hot rolling it is necessary to have the closed porosity. It is possible if the relative density of the samples will be more than 90 %. So at the first step the cold rolling had been used.

During the cold rolling the degree of compression of sintered materials depends upon their height. Samples with the height of 36 mm require the degree of compression not more than 10 % of their height. If the degree of compression will exceed 12 % we will observe the formation of macroscopic cracks in the middle part of the sample and there are parallel to the surface of compression (Fig. 1), because the samples after sintering are characterized by alternative porosity with the maximum value of porosity in this zone. For the samples with lower height with the density more than 90 % destruction occurs only for the compression ratio more than 30 %.

Fig. 1. Common view of the billet after compression more than 10 % during the rolling process

The investigation of sample microstructure under the ultimate deformations showed the formation of large number of plane microscopic cracks (Fig. 2). The process of destruction takes place in the whole sample volume. So depending on sample size, kind of rolling machine, roller diameter and the speed of rolling the optimal values of compression ratio should be determined after sintering and after the following steps of annealing.

For the following rolling steps of the samples with closed porosity the hot rolling had been tested. Corresponding experiments had shown that hot rolling resulted to the development of the formed cracks (Fig. 3).

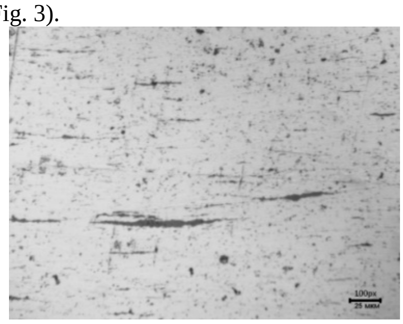


Fig. 2. Microstructure of the material after compression more than 30 % during the rolling

Fig 3. Common view of the billet after compression more than 10 % during hot rolling at 1100 ^{o}C

The following tests showed that required parameters of rolled samples can be obtained by means of cold rolling with compression ratio not more than 10 % with the following annealing at the temperature not less than 1200 ^{o}C. The initial annealing should be performed in protective environment (inert gas or vacuum). At the next steps it is possible to perform the annealing in air environment. The samples after cooling in air have the black color due to the formation of spinel which prevents further rolling. During the heating in vacuum spinel decomposes and at the surface of the sample pure chromium oxide is formed. So it is reasonable to use annealing in vacuum at all stages of rolling process.

After the sintering or annealing in vacuum furnace during two hours the samples had been cooled in air with the formation of solid solution during the quenching process. X ray investigations confirm the structure of ideal solid (Fig. 4).

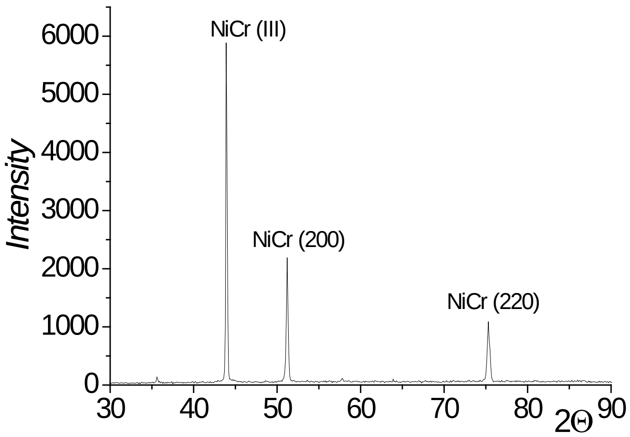
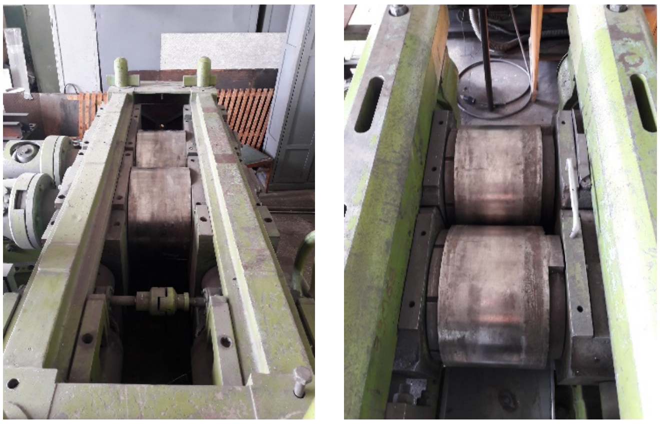


Fig. 4. X-Ray diagram for disperse-hardened nichrome based alloy

By means of the experiments the following rolling regimes for rolling unit DUO 500 (Fig. 5, a) at room temperatures for developed alloys for the thicknesses from 20 mm till 3,5 mm had been established: 5 cycles – rolling along the samples, deformation per one pass 2-3 % and the last rolling cycle should be performed in transverse direction. Intermediate annealing should be performed at the temperature 1200 ^{o}C during 2 hours in vacuum furnace after every cycle of rolling.

At the unit DUO 300 (Fig. 5, b) it is possible to obtain the sizes from 3,5 mm till 1,8 mm and at the unit Qvadro 160/100 (Fig. 6) it is possible to obtain the sizes from 1,8 mm till 0,3 mm.



a b

Fig. 5. Rolling unit DUO 500 (a) and DUO 300 (b) (IPMS NASU)

After three cycles of cold rolling with intermediate annealing at the temperature 1200 ^{o}C porosity decreased up to 7,3-7,4 %. During the following rolling cycles porosity decreased too and required value about 3 % can be reached after the fifth cycle. Corresponding values of alloy density in that case are equal 7450-7500 kg/m^3.

Fig. 6. Rolling unit «Qvadro 160/100» (IPMS NAS of Ukraine)

It should be noted that the sheets of the alloys with the thickness less than 0,3 mm had been produced on rolling unit Mill DUO-280 at the Rubin company, Kharkov, Ukraine. (Fig. 7).

According to practical requirements (the calculations of the specialists from SDO Yuzhnoye) the sizes of the billets for the production of heat protection system model should be equal (0,15-1,0) x150x150 mm. To ensure such sizes using above described equipment it is necessary to produce by pressing the samples with the thickness up to 12 mm. It is not difficult to calculate that for the production of the billet with the thickness 0,4 mm from initial sample with the thickness 12 mm using intermediate annealing in vacuum at the temperatures 1200 ^{o}C during one hour and compression ratio not more than 10 % it is necessary more than 40 working days.

Fig. 7. Rolling unit Mill DUO-280 (Rubin company, Kharkov)

It should be noted that in parallel with the production of billets it was necessary to make the modernization of the rolling machine to have a possibility of realization of the full cycle of rolling operations that had been developed in IPMS NASU for such kind of heat resistant nichrome based alloys. Developed technology allows decreasing of the time of billet production for heat protections models up to 10 times due to the possibility of rolling of the sheets with the thickness of about 1 mm and in that case the required size of billets of about 0,3 mm can be obtained much faster.

4. Physical and mechanical properties of nichrome based alloys

Physical and mechanical properties of developed nichrome based alloys had been investigated at the temperature interval of 20-1200 °C (table 1). It was established that its tensile strength limit at room temperatures is equal 1020 MPa. Its specific strength at room temperature exceed the corresponding value for developed earlier alloy ЮИПМ-1200 [6] of 1,5 times and at high temperatures the enhancing of two times had been obtained.

Table 1. Physical and mechanical characteristics of nichrome based alloys

Property	T, °C	ЮИПМ-1200	Developed alloy
Density		8300	7900
Tensile strength limit, $\sigma_в$, MPa / MPa/kg	20	738/0,09	1020/0,13
	800	237/0,03	542/0,07
	1100	45	-
	1200	-	40
Yield strength, $\sigma_{0,2}$, MPa	20	364	624
	800	228	457
	1100	40	-
	1200	-	35
Relative elongation, δ, %	20	36,5	21
	800	36,7	18,1
	1100	32,8	18
	1200	-	18
Relative compression, ψ, %	20	34,2	22
	800	32,6	19
	1100	19,0	-
	1200	-	20

5. High temperature tests of the developed nichrome based alloy at the special stand in IPS NASU

To test the efficiency of developed alloys at the conditions simulating the service ones and to evaluate the possibility of using of such stand for the further tests of thermal protection system model the experiments on thermal and mechanical resistance of materials in high temperature gas flows had been performed at gas and dynamic stand of Pisarenko Institute for Problems of Strength of National Academy of Science of Ukraine [8-10]. Test cameras of gas dynamic stand and common view of this stand during the experiments are presented at fig 7 and fig.8. The sample of alloy before testing is presented at fig.9.

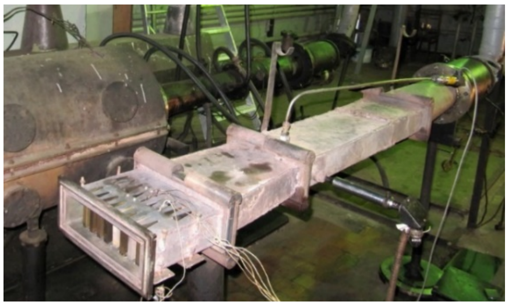


Fig. 7. Cameras of gas and dynamic stand

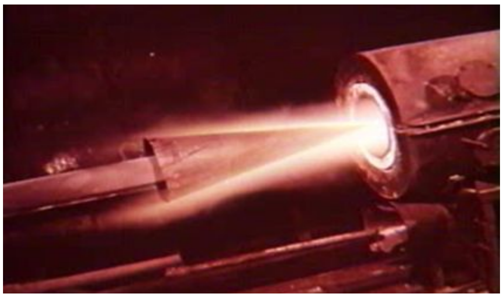
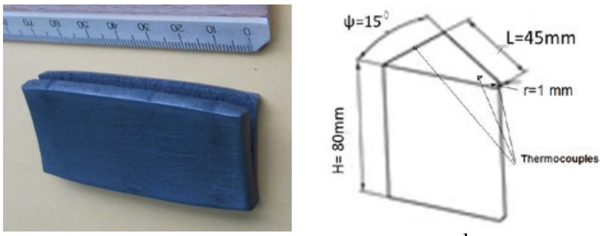


Fig. 8. Tests on gas and dynamic stand



a b

Fig. 9. Sample of alloy before tests (a) and witness sample (b)

The sample from developed alloy had been tested under the thermal loadings simulated the working conditions of heat protection systems in correspondence with given program. The changing of the temperature for gas flow (1) and witness sample (2) and also the temperature of gas flow simulating the working conditions of heat protection system (3) during the performed tests are presented on fig.10. Changing of the temperature for the witness sample and for the sample from developed alloy during the cycle of thermal loading are presented on fig. 11.

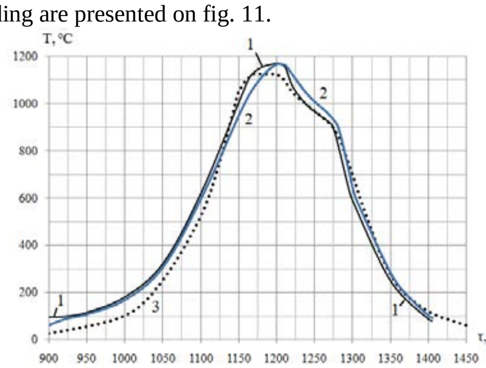


Fig. 10. Temperature changes for:
1 – gas flow; 2 – edges of witness sample;
3 – flow during service (calculated value)

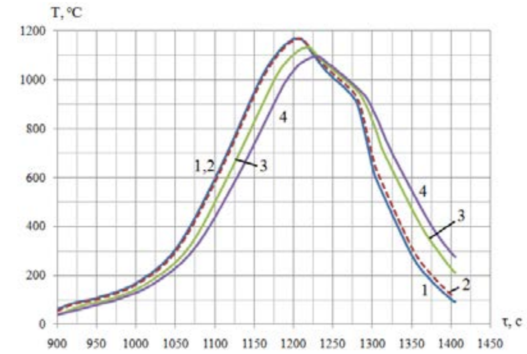


Fig. 11. Temperature changes during the cycle:
1 – edge of witness sample; 2 – edge of powder alloy and witness sample at the distance of 3 mm from the edge; 3 – back side of witness sample; 4-powder alloy sample

To test the influence of high temperature gas flow on the developed alloy its macro and micro hardness had been determined before and after high temperature tests

Macro hardness had been studied using portable hardness tester COMPUTEST SC produced by ERNST company (Switzerland). Rockwell hardness had been investigated in correspondence with the scale C (HRC). Loading on the diamond indenter in the form of cone with the top angle of 100^0 was equal 49 N. The measurements of micro hardness had been made on the ПМТ-3 machine for the micro sections with indenting of diamond tetrahedral pyramid at a load of 100 g (table 2).

Table 2. The values of Rockwell macro- and micro hardness before and after tests.

Rockwell hardness, HRC scale		Microhardness, H_μ, GPa	
Before tests	After tests	Before tests	After tests
35,7	33,4	3,06	2,83

It should be noted the slight decreasing of hardness values (33,4 HRC) in comparison with corresponding values after high temperature tests (35,7 HRC) which was equal of about 6 -7 %.

Micro hardness of tested samples had been equal ~3 GPa. Changing of micro hardness values for the experiments before and after high temperature tests was equal 6-7 %. It changing is within the range of the data scatter, which is due to the process which takes place after deformation hardening.

Analysis of obtained results had shown that slight decreasing of macro- and micro hardness (6-7 %) is an evidence of saving of strength properties of developed materials after double exposure in a high-temperature gas flow..

So the developed material meets the requirements of thermal resistance in high temperature gas flow and it can be recommended and used for the production of heat resistant thermal protection systems constructions.

6. Conclusions

1 Rolling regimes for dispersed hardened powder nichrome based alloy had been developed. These regimes allow to produce the sheets of such alloy with the thickness from 0,6 mm till 0,4 mm and the size of 150x150 mm necessary for production of the heat resistance system model.

2 Physical and mechanical properties of developed alloy had been developed. It was found the following:

- alloy density is equal 7450-7500 g/cm^3;
- tensile strength limit at room temperature is equal 1020 MPa and at 800 ^{o}C corresponding value is equal 542 MPa and its specific strength at this temperatures exceed corresponding values for YuIPM-1200 alloy developed earlier on 1,5 times at room temperatures and on more than 2 times at high temperatures;
- alloy has sufficient plasticity so it may be processed by pressure.

3 High temperature tests of developed alloys had been made according to the special program at gas and dynamic stand at IPS of NASU. This program simulated the service conditions of heat protection systems. These tests showed that developed alloy has required level of thermal resistance in high temperature gas flow and it can be used for production of heat resistance constructions of thermal protection systems.

4 It was established that at present time in Ukraine gas dynamic stand situated in IPS of NASU is the most reliable for testing of real models of heat protection systems using given program.

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