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Warm upsetting tests with cylindrical molybdenum and wolfram samples

ABSTRACT

The resistance of a modern ceramic material – silicon nitride – was examined by cyclical high temperature and compressive loads by the use of warm upsetting tests at cylindrical molybdenum and wolfram samples. Furthermore, flow curves of these samples were determined for different temperatures.

Keywords: *upsetting test, silicon nitride, molybdenum, wolfram, flow curves*

1. INTRODUCTION

The suitability of new developed silicon nitride (Si_3N_4) qualities for the use as rolls and other components for the rolling technology was introduced in previous publications [1, 2]. These studies were carried out in upsetting tests, where upsetting tools made of different silicon nitride qualities have been used. Cold and warm upsetting tests were done with cylindrical steel and aluminum samples (Ø 20 mm, H = 40 mm), copper-tin (CuSn) and copper-silver (CuAg) samples (Ø 4 mm, H = 8 mm). In other upsetting tests, samples made of steel S235JR were determined at a temperature of 800 °C and an upsetting force of 600 kN.

The experimental studies for cold and warm rolling were carried out with Si_3N_4/steel compound rolls in a duo roll stand with a coiler. A steel wire (D9-1) with a diameter of 4 mm was rolled to a flat wire with a thickness of 0.6 – 0.8 mm [2].

The results of industrial trials of Si_3N_4 work rolls for cold rolling of thin foils (foil thickness up to 0.05 mm) from stainless ferritic steels as well as austenitic steels in a 20-roll-Sendzimir stand can be found in additional publications [3].

The aim of the present study is to verify the performance of silicon nitride with practical metal forming orientation and to determine, if the examined material can with stand high cyclical temperature and compressive loads.

Another aim of this work is the determination of flow curves of compressed molybdenum and wolfram samples under practical forming conditions at different temperatures with upsetting tools made of silicon nitride.

2. EXPERIMENTAL INVESTIGATIONS AND RESULTS

High-quality silicon nitride ceramic samples with a diameter of 50 mm and a height of 30 mm were manufactured in a HIP-process (hot isostatic pressing at a sinter pressure of 2000 bar). The surface of the samples had a roughness of Ra = 0.13 µm after finishing (Fig. 1).

Figure 1. Silicon nitride sample

Slika 1. Primer silicijum nitrida

The samples were embedded in matrices made of a steel 100Cr6 (shrink temperature 250 °C) (Fig. 2) by a hydraulic press. Supporting plates of carbide were used.

The warm upsetting tests were carried out with cylindrical samples made of molybdenum and wolfram with a diameter of 20 mm and a height of 40 mm. Fig. 3a shows the samples before heating in a furnace.

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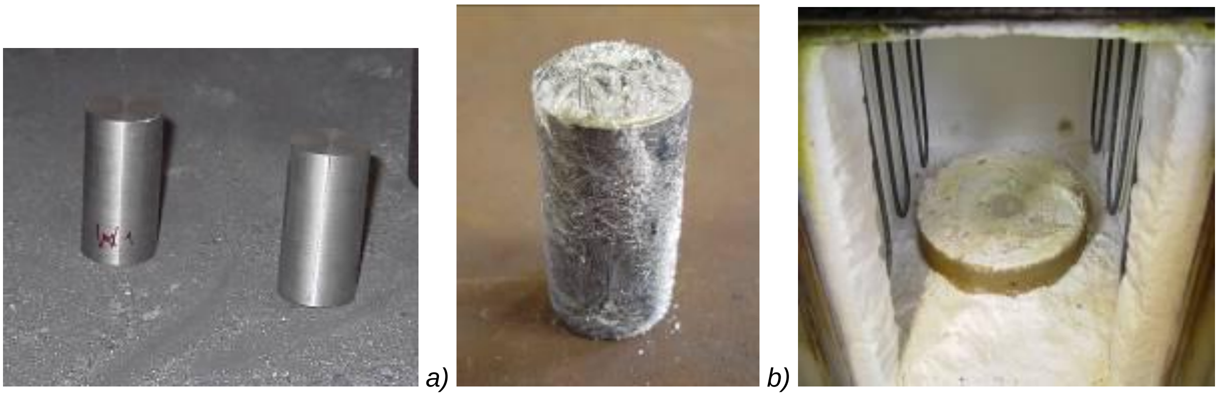
Figure 2. Si_3N_4 test samples

Slika 2. Si_3N_4 test uzorak



a) b) c)

Figure 3. Heating of wolfram and molybdenum samples in a furnace: a – samples before heating; b – molybdenum sample after heating up to 1600°C without protective gas; c – interior of the furnace after heating up of a molybdenum sample without protective atmosphere

Slika 3. Zagrevanje uzoraka volframa i molibdena u peći: a) uzorci pre grejanja; b) uzorak molibdena posle grejanja na 1600°C bez zaštitne atmosfere; c) unutrašnjost peći nakon zagrevanja uzorka molibdena bez zaštitne atmosphere

The molybdenum sample has formed an oxide layer with a needle-like structure after heating up to a temperature of 1600 °C without protective atmosphere in the furnace (Fig. 3b).

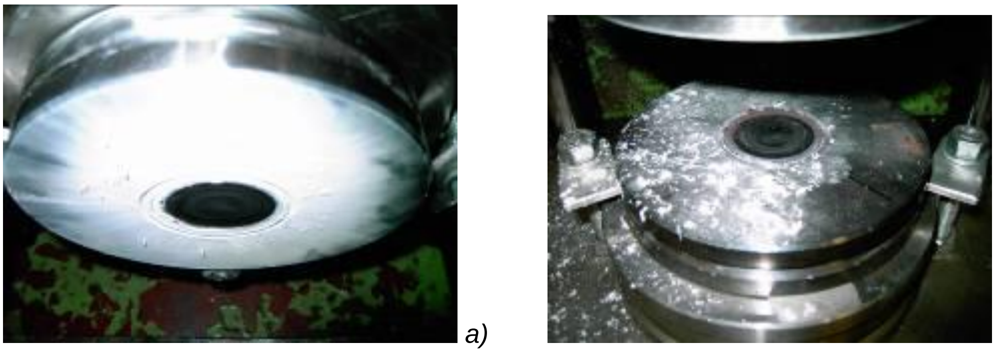
Wolfram is chemically stable at room temperature in air and water and shows oxidation when it is heated up. A noticeable oxidation starts at a temperature of about 400 – 600 °C. An additional increase of the temperature leads to an intensive oxidations with formation of WO_3 with an exponential development. At temperatures above 1000 °C WO_3 starts to vaporize. The vaporization and formation speed of WO_3 is equal at a temperature of 1250 °C [4]. Fig. 3c shows the interior of the furnace after heating up of the wolfram sample without a protective atmosphere. The thick snow-like WO_3 deposit is clearly visible.

The heated wolfram sample was compressed with a force of 1400 kN. The heat produced by the compression process leads to a sample heating and as a result to vaporization of WO_3 and additional oxidation of the sample. Strong deposits of the vaporized WO_3 could be determined at both upper and lower tools (Fig. 4).

An intensive oxidation of the high-temperature melting metals during heating leads to an absorption of harmful gases, which decreases the mechanical properties of the rolled stock. A high efficient protective measure of oxidation and gas absorption would be heating, rolling and cooling down in vacuum or under a protective atmosphere [5].

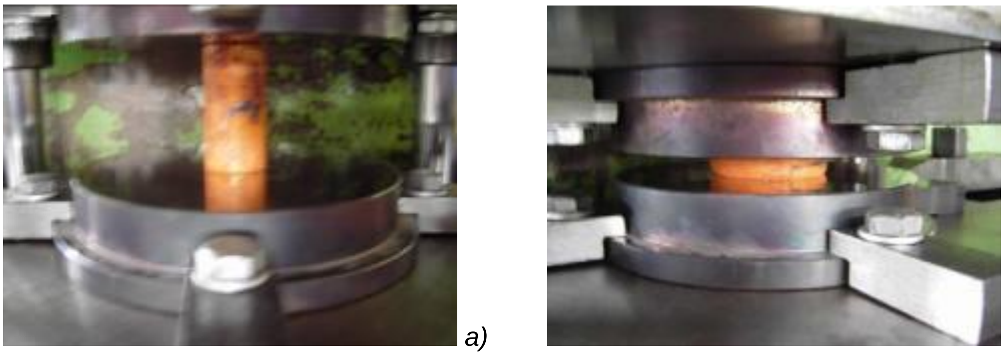
To prevent the effects mentioned above, both wolfram and molybdenum samples were heated up in the furnace in a protective atmosphere (argon) up to temperatures of 1350 °C and 1600°C. The samples were compressed with a force of 1400 kN and a deformation rate of 10-15 cm/min up to degrees of deformation φ = 1.4 – 1.6 (Fig. 5).

During compression of the molybdenum and wolfram samples, a combination of compressive and shear stresses with simultaneous high thermal loads are acting to the silicon nitride upsetting tools. At the end of the tests the surfaces of the compression tools were free of micro and shear cracks and showed no disruption of the surface (Fig. 6).



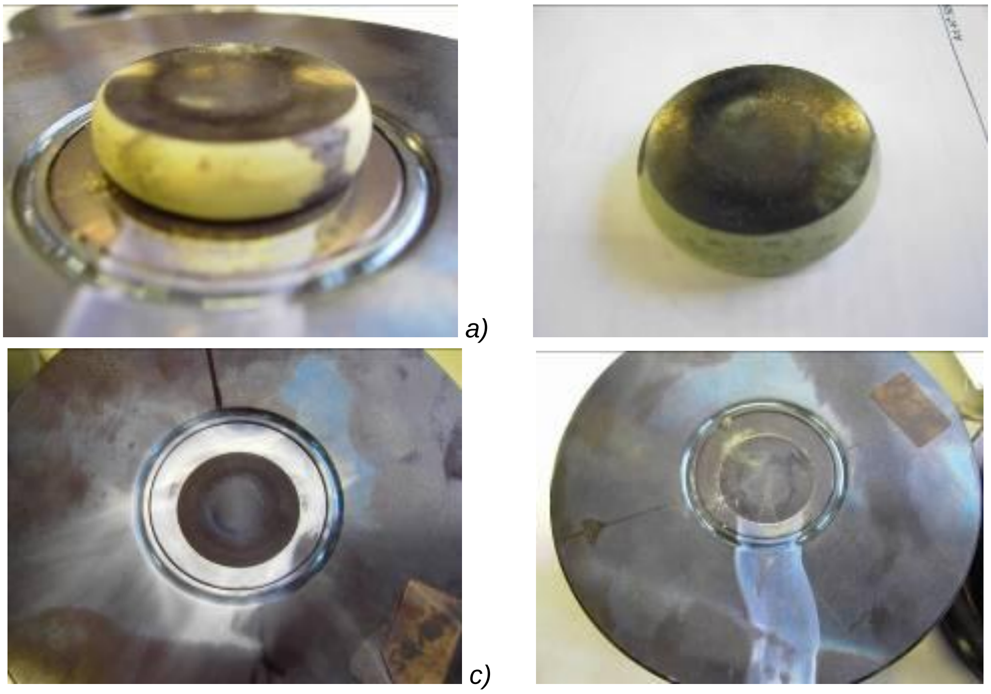
a) b)

Figure 4. Compression tools made from silicon nitride after warm upsetting tests with cylindrical wolfram samples heated up without protective atmosphere: a) upper tool, b) lower tool
Slika 4. Alati za kompresiju napravljeni od silicijum nitrida nakon toplih testova pobude sa uzorcima cilindričnog volframa zagrejanih bez zaštitne atmosfere: a) gornji alat; b) donji alat



a) b)

Figure 5. Hydraulic press with compression unit and warm compressed cylindrical sample: a) start of test, b) end of test
Slika 5. Hidraulična presa sa kompresijom i toplo komprimovani cilindrični uzorak a) početak testa, b) kraj testa



a) b) c) d)

Figure 6. Compressed samples (a, b) and compression tools made of silicon nitride after warm upsetting tests with cylindrical molybdenum and wolfram samples: c) upper tool, d) lower tool
Slika 6. Kompresovani uzorci (a, b) i alati za kompresiju napravljeni od silicijum nitrida nakon ispitivanja toplog uzbuđenja sa cilindričnim molibdenom i uzorcima volframa: c) gornji alat, d) donji alat

No adhesion of the samples could be determined at the compression tools. Therefore it can be assumed, that the tested Si_3N_4-material can withstand the cyclic appearing temperature and compression loads. For a consistent discussion of the results, additional tests are necessary. Figure 7 shows the flow cure of the molybdenum compression samples for different temperatures.

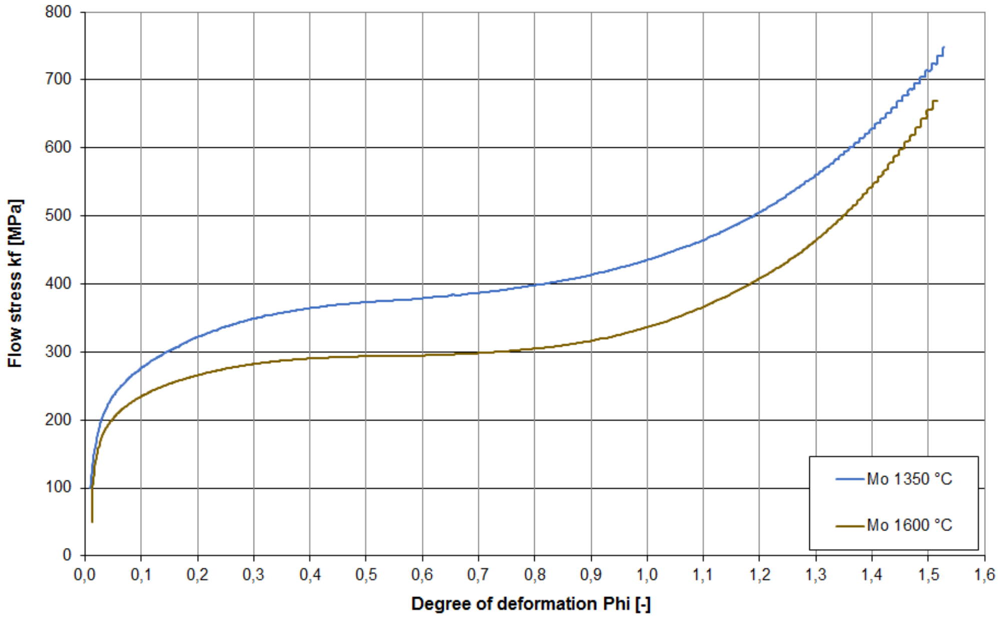


Figure 7. Flow curves of cylindrical molybdenum samples at 1350°C (with protective gas) and 1600 °C (without protective gas)

Slika 7. Krive protoka cilindričnih uzoraka molibdena na 1350°C(u zaštitnoj atmosferi) i na 1600°C(bez zaštitne atmosfere)

An effect of the heating becomes apparent. At low degrees of deformation at the beginning of the compression tests, the slope of the flow curve is almost similar at both temperatures. The flow stress, which is necessary for the plastic deformation of the sample, is lower at higher temperatures. At further plastic deformation, this effect is more recognizable and a difference of the flow stress of about 100 MPa can be seen in the entire course of the flow curves. The force that is necessary for the plastic deformation at the same degree of deformation is decreasing with increasing temperatures. At a degree of deformation of φ = 1 the material shows a strong hardening effect. This leads to a recognizable increase of the flow stress up to a maximum of 750 MPa at compression temperatures of 1350 °C resp. 670 MPa at 1600 °C. At both temperatures the maximum degree of deformation of the molybdenum samples was about φ = 1.5.

Figure 8 shows the flow curves of the wolfram samples compressed at a temperature of 1600 °C.

The effect of a protective atmosphere in a furnace to the flow stress was examined in these tests. The atmosphere has a significant effect to the surface quality of the samples. The needle-like oxide layer of the wolfram sample, which arises in the furnace without a protective atmosphere, leads to a reduction of the flow stress. The difference of the flow stress at same degrees of deformation is about 100 MPa. The oxidized sample has a higher maximum degree of deformation and the flow stress reaches a maximum comparable to the sample that was heated up in a protective atmosphere. From the metal forming point of view the oxidized sample is easier to deform plastically, but is unusable due to the bad surface quality of both, samples and tools. Therefore, the use of a protective atmosphere is indispensable for the plastic processing of this material.

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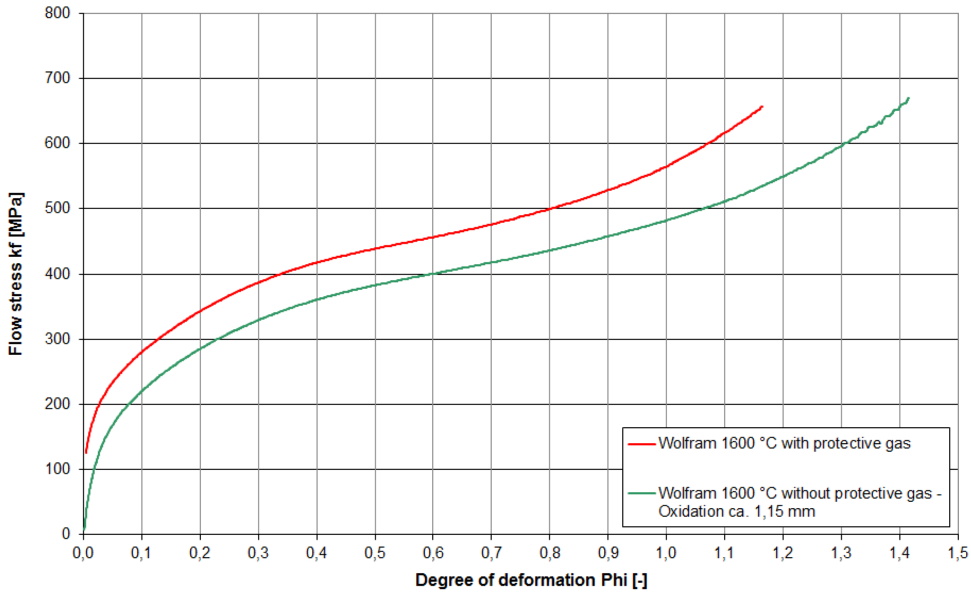

Figure 8. Flow curves of cylindrical wolfram samples at a temperature of 1600 °C: green: heating without protective gas, red: heating with argon atmosphere

Slika 8. Krive protoka cilindričnih uzoraka volframa na temperaturi od 1600°C: zeleno: grejanje bez zaštitnog gasa; crveno: grejanje argonom

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IZVOD

TESTOVI ZA POBUĐIVANJE TOPLOTE SA CINLINDRIČNIM MOLIBDENOM I VOLFRAM UZORCIMA

Otpor savremenog keramičkog materijala - silicijum nitrida - ispitivan je cikličnim visokim temperaturama i pritiscima na korišćenje testova toplog uzbuđenja na cilindričnom molibdenu i uzorcima volframa. Pored toga, krive protoka ovih uzoraka su određene za različite temperature.

***Ključne reči**: test pobude, silicijum nitrid, molibden, volfram, krive protoka*

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