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STUDY ON THE PHASE COMPOSITION AND MICROSTRUCTURE OF Ti-8Al-1Mo-1V AFTER SURFACE PLASMA NITRIDING

The application of titanium alloys is limited due to their low surface hardness and wear resistance, especially for parts operating under friction and contact loads. One of the most widely used technologies for the thermochemical treatment of titanium alloys is gas nitriding. A new method in this direction is surface plasma gas nitriding using indirect arc plasmatrons operating in a chamber with a controlled nitrogen atmosphere. In the present work, the changes in the phase transformations, microstructure, and surface hardness of titanium alloy Ti-8Al-1Mo-1V after plasma gas nitriding at the power of 18 kW, and 25 kW for a time between 5 and 30 minutes are studied. The plasma gas nitriding with the indirect plasmatron of the titanium alloy produced continuous surface layers. Analysis of the surface showed the presence of TiN and TiO₂. The thickness of the plasma gas nitrided layers ranges between 100 μm and 350 μm, depending on the technological parameters.

Keywords: plasma spray; nitriding; indirect plasmatron; surface treatment; Ti-8Al-1Mo-1V

1. Introduction

The use of titanium and its alloys for the production of various components and parts in various industries has been a clear trend in recent years. Despite their excellent set of properties including high specific strength, good high-temperature mechanical properties, and high corrosion resistance the application of these alloys is limited by their low surface hardness and wear resistance, especially for parts operating under friction and contact loads. To overcome some of these disadvantages various surface hardening techniques can be employed. One of the commonly used techniques for the surface hardening of titanium alloys is nitriding [1-7].

Regarding the technologies and techniques for nitriding, a relatively new method in this direction is surface plasma gas nitriding using indirect arc plasmatrons operating in a chamber with a controlled nitrogen atmosphere. The formation of a nitride layer on titanium surfaces, during plasma gas nitriding, using indirect plasmatron is a rather complex thermochemical diffusion process. In general, when the titanium alloy sample is exposed to a high-temperature active nitrogen media (the average temperature of the plasma jet is in the range of $17-18 \times 10^3$ K), nitrogen diffuses into the base, thus forming various nitrogen compounds. As a result of this thermochemical diffusion, a surface layer

composed of nitrides is formed. The color of the nitrided titanium surfaces is related to the employed technological parameters of the regime, and the newly formed phases [8-13].

Ti-8Al-1Mo-1V is near alpha titanium alloy. It consists of 8% of aluminium as an alpha stabilizer and a few additions of molybdenum, vanadium, and iron, that form a small amount of beta phases. This alloy was designed to provide good creep resistance and it serves this purpose well up to 450°C. Amongst all of the commercially available titanium alloys, Ti-8Al-1Mo-1V has the highest tensile modulus and lowest density. It has been designed particularly for applications in the aerospace industry. It is usually used but is not limited to the production of aero-engine parts such as forged fan and compressor blades, spacers, and seals.

The present work explores the changes in the phase transformations, microstructure, and hardness of the titanium alloy Ti-8Al-1Mo-1V after plasma gas nitriding at various power, for different time intervals are studied.

2. Experimental

For this study, samples of titanium alloy Ti-8Al-1Mo-1V undergo thermochemical surface treatment using a nitrogen plasma stream jet generated by an indirect plasmatron with a power

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of 18 kW, and 25 kW. First, the specimens for nitriding were cut to dimensions of 10 mm × 10 mm × 4 mm. The characteristics and chemical composition of the titanium alloy samples are according to [14]. Before the nitriding process, several pretreatment operations were performed on the samples including mechanical grinding and polishing. In this research, a PPN 800 plasma spray apparatus equipped with an indirect plasmatron is used. The thermochemical surface nitriding is carried out in a chamber with a controlled nitrogen atmosphere. The nitrogen gas used for the process has a purity of 99.998%. The overpressure of the atmosphere in the chamber is maintained with a constant nitrogen flow at a flow rate of 100 ml/min. The distance from the tip of the plasmatron nozzle to the sample was 120 mm and it was kept constant for all of the conducted experiments. During the nitriding process, the treated samples were firmly fixed and motionless. The plasmatron traverses on the surface of the sample in a reciprocating motion at a speed of 4 meters per minute. The duration of nitriding process was carried out for different periods of time between 5 and 30 minutes. In the course of the thermochemical treatment with a nitrogen stream jet of plasma, the surface layer of the samples did not melt. The following techniques and equipment were used for the characterization of the acquired surfaces: an optical microscope equipped with a digital camera and software (LM-308); an X-ray diffractometer (Philips X Pert PRO analytical, using Cu-K α radiation); and a microhardness tester (DVK 1-AT).

3. Results and discussion

In the employed nitriding process, the stream jet of plasma brings atomic and ionized nitrogen to the surface of the exposed titanium samples at a speed of 500 m/s. Depending on the power of the plasmatron in the studied range the temperature on the surfaces of the experimental specimens elevates until it reaches values up to 1100°C [8]. Under those conditions, nitrogen diffuses into the samples of titanium alloy Ti-8Al-1Mo-1V forming various nitrogen compounds.

During the nitrogen diffusion, on the surface of the samples, a thin brittle compound layer that consists of titanium nitrides and titanium oxides is formed with a subsequent diffusion zone below it. It was found that the thickness of the surface compound layer grows from 4 μ m to 10 μ m within the limits of the used regimes in this study. Moreover, during the thermochemical treatment, the compound layer most probably pulverizes and separates from the surface under the influence of the plasma stream jet and arising internal stresses (Fig. 1). Nevertheless, its rapid formation prevents the growth of a deep diffusion layer.

3.1. Phase analysis

As a visual indicator for structural changes following the formation of new phases may be taken into consideration that the surface layer changes its color during the plasma nitriding

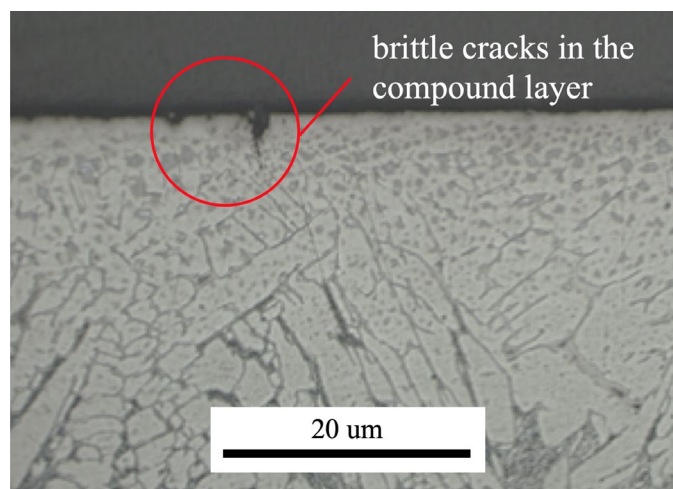


Fig. 1. Cross section of Ti-8Al-1Mo-1V sample after plasma gas nitriding at 25 kW power for 30 minutes

process. Initially, the surface of the Ti-8Al-1Mo-1V samples is gray after 5 minutes of the thermochemical treatment using both 18 kW and 25 kW of power. From about 6 to 10 minutes of nitriding, the surface changes its appearance to orange-brown. Furthermore, between 10 to 15 minutes it acquires a red-orange hue, and from 15 to 30 minutes of processing time, the surface color has a brownish tint. This is also confirmed by the X-ray analysis. The results for samples nitrided at a power of 18 kW are shown in Fig. 2, and Fig. 3 displays analysis for specimens nitrided at 18 kW and 25 kW of power for 30 minutes.

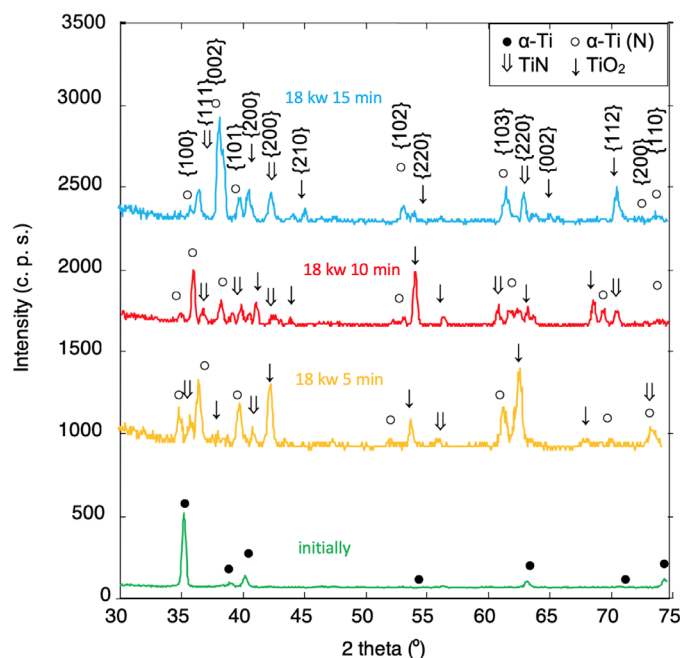


Fig. 2. Diffraction pattern and X-ray phase analysis of Ti-8Al-1Mo-1V after plasma gas nitriding at 18 kW power for 5, 10, and 15 min

The conducted phase analysis showed that initially the surfaces of the tested Ti-8Al-1Mo-1V samples were composed of α -Ti (Fig. 2). As a result of the plasma gas nitriding process,

the α -Ti is converted to hexagonal close-packed (hcp) α -Ti(N), face-centered cubic (fcc) TiN, and tetragonal TiO₂ crystal structures. The α -Ti(N) phase differentiates from the α -Ti because of the peaks which belong to the hexagonal structure, that move to the lower 2θ angles, leading to an increase in the lattice parameters due to the interactions with nitrogen. The presence of oxygen is not excluded, but it is expected to be considerably less than nitrogen.

In all experiments, we have registered the presence of the tetragonal TiO₂ phase along with the TiN in the surface nitrated layer. The main reason for the detected TiO₂ might be that despite the high purity of the nitrogen used in the process, it is possible to have minimum but sufficient amounts of oxygen in the gas bottles. The formation of the oxide film can also be explained by the reasonably high temperatures on the sample surface, typically above 800°C, where the oxygen activity is very high.

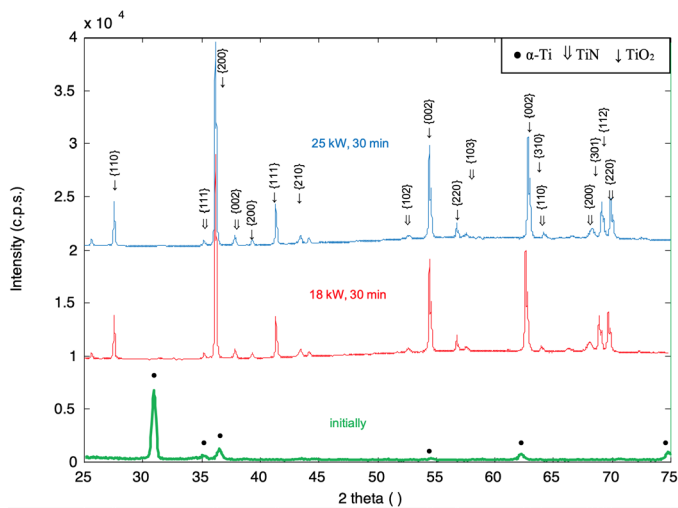


Fig. 3. Diffraction pattern and X-ray phase analysis of Ti-8Al-1Mo-1V initially, after plasma gas nitriding at 18 kW, and 25 kW power for 30 min

3.2. Microstructure

The microstructure of the nitrated Ti-8Al-1Mo-1V samples is displayed in Fig. 4. There are three distinctive zones formed during the nitriding process: a mixture of different phases (compound layer), the diffusion zone, and the base material. In most of the samples, there is a clear boundary between the compound layer and the diffusion zone. The compound layer covers the entire surface of the sample. It is hard, thin, and fragile. Its thickness varies from 4 to 10 μ m. Beneath the compound layer is the diffusion zone. By prolongation of the processing time in the studied interval, and with the increase of power from 18 kW to 25 kW, the thickness of the nitrated layers grows.

Furthermore, there are substantial differences in the microstructure of the plasma gas nitrated Ti-8Al-1Mo-1V samples at 18 kW and 25 kW as can be observed in Fig. 3. The microstructure for the plasma gas nitrated samples at 18 kW is homogeneous, while it appears to be heterogeneous in samples nitrated at 25 kW. The size of the titanium nitride grains, formed in the diffusion zone using 18 kW of power is much smaller. Most probably, this difference is due to the higher temperature that occurs using a power of 25 kW. By using that amount of power, the surface temperature of the treated samples exceeds the β -transus.

For all the samples, plasma gas nitrated with an indirect plasmatron in a chamber at a power of 25 kW the diffusion layer may be subdivided into two sublayers – outer and inner. The outer sublayer is right under the compound layer. It is a continuous α -Ti solid solution enriched with nitrogen. In most cases, this is a uniform white layer formed during the plasma gas nitriding process. The thickness of this layer grows with the increasing nitriding time. The inner sublayer, which is between the outer sublayer and the unreacted base material, consists of white coarse-grained columns and a fine structure resembling the base layer. These coarse grains, grown in depth, are usually

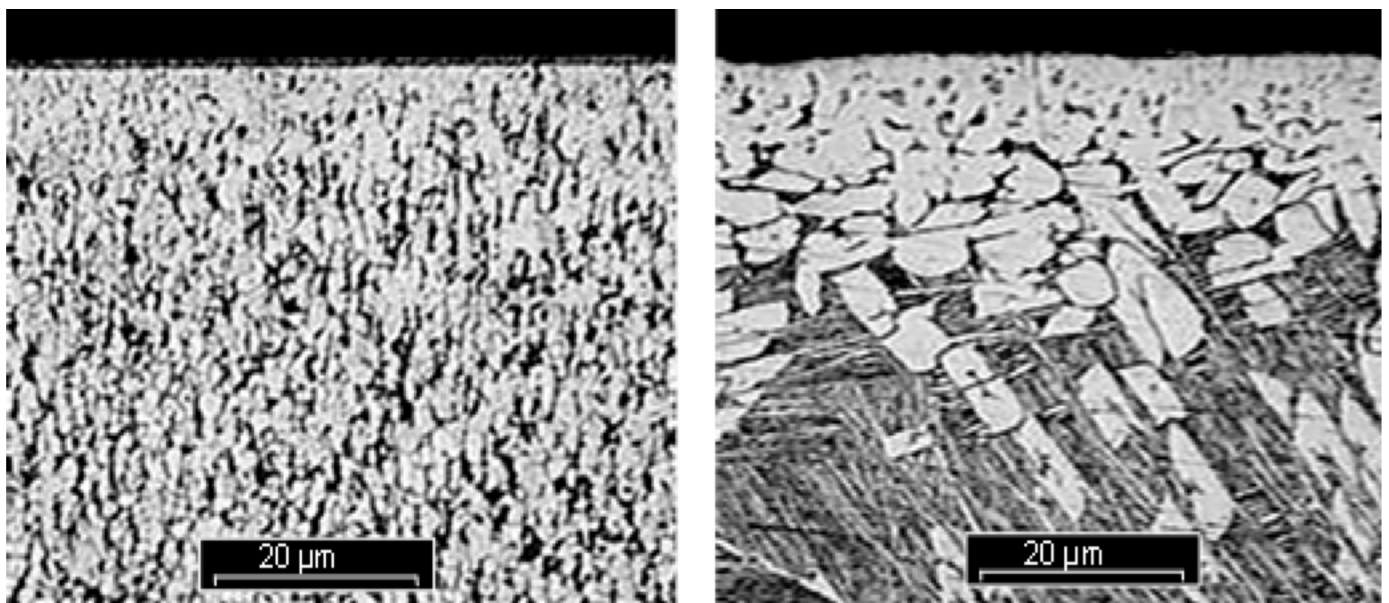


Fig. 4. Microstructure of Ti-8Al-1Mo-1V after plasma gas nitriding at 18 kW (left) and 25 kW (right) for 15 minutes

inclined, and in some places are at the boundaries of the basal grains. Considering that the temperature of the plasma gas nitrated samples at 25 kW most certainly exceeds the β -transus, the formation of this layer is most likely to occur during the phase transformation β -Ti(N) \rightarrow α -Ti(N). The β -Ti enriched with nitrogen phase transforms into α -Ti(N, O) phase when the temperature decreases. Furthermore, the α -Ti(N, O) phase can release titanium nitrides upon further temperature decrease. It is well established that nitrogen solubility is much lower in β -Ti than in α -Ti. Therefore, the microhardness values in the inner sublayer are expected to be considerably lower than those in the outer sublayer of the diffusion zone.

As the power rises from 18 kW to 25 kW, the thickness of the nitrated layers grows. This can be explained by the diffusion coefficient, which increments with the temperature elevation. The nitriding time in the studied interval has a similar effect on the nitrogen diffusion, and the thickness of the layer respectively. The maximum thickness of the plasma gas nitrated layer achieved within the limits of this study varies between 100 μm and 350 μm . That also can be identified by the microhardness cross-section profiles.

3.3. Microhardness

The microhardness of the plasma gas nitrated specimens was measured on the surface and on cross-section to assess the effect of the nitrogen-diffusion hardening. The acquired data was evaluated and the microhardness profiles are presented in Fig. 5 and Fig. 6.

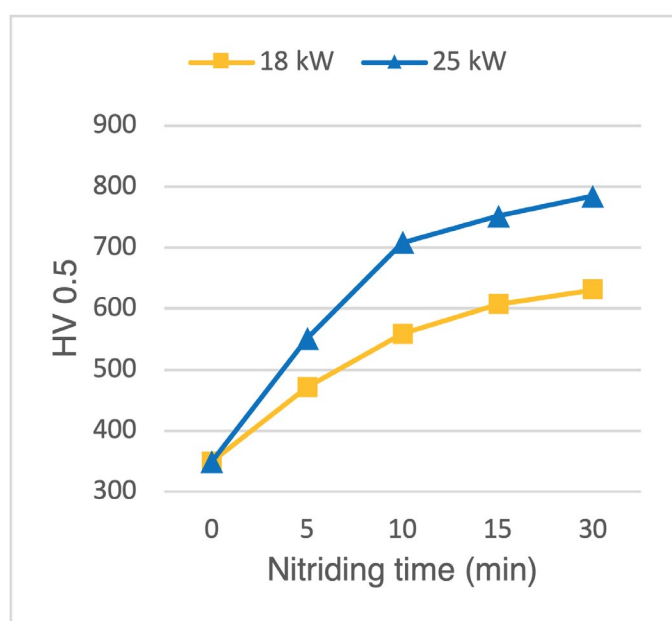


Fig. 5. Microhardness profiles of Ti-8Al-1Mo-1V after plasma gas nitriding with indirect plasmatron using 18 kW, and 25 kW (average values)

From the data available in Fig. 4, we can conclude that the microhardness in the studied interval increases with power

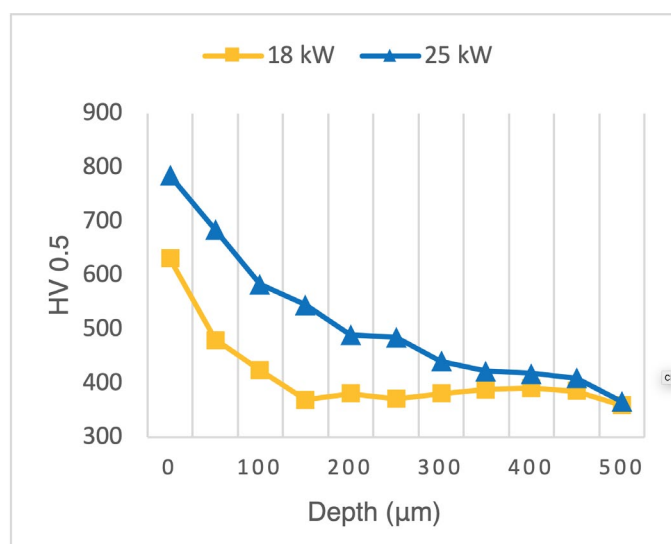


Fig. 6. The influence of power and treatment time on the microhardness of Ti-8Al-1Mo-1V after plasma gas nitriding with indirect plasmatron

and nitriding time. However, as the microhardness increases, so does the brittleness of the surface layer. The elevated hardness of the plasma gas nitrated samples is a result of the formation of TiN during the nitriding process. Thus, by measuring the microhardness in points on a cross-section we can determine the thickness of the nitride layer, and gather information about the nitrogen diffusion.

By observation of the microhardness profiles displayed in Fig. 5, we can track the nitrogen diffusion and measure the thickness of the nitrated layer. The hardness values of the surface layers are considerably higher (over 800 HV using 25 kW power). Further, in the diffusion zone, the microhardness decreases, when it reaches the values of the base material. The thickness of the nitrated layers varies from 100 μm to 350 μm . That also corresponds to the measurements from the micrographs. The thickness of the nitrated layer grows with the power from 18 kW to 25 kW.

4. Conclusion

Specimens of titanium alloy Ti-8Al-1Mo-1V were plasma gas nitrated with indirect plasmatron using 18 kW, and 25 kW of power for 5, 10, 15, and 30 minutes. The phase composition of the thermochemically treated layer after plasma gas nitriding consists of α -Ti(N), TiN, and TiO₂. Analysis of cross sections shows the presence of three distinctive zones: compound layer, diffusion zone, and base metal. On the outermost surface of the nitrated samples is a compound layer, which consists of TiN and TiO₂. Its thickness varies from 4 μm to 10 μm within the limits of the regimes employed in this work. Diffusion zone forms between the compound layer and the base metal. It comprises of α -Ti solid solution enriched with nitrogen. The thickness of the compound layer and the diffusion zone is measured based on the microhardness as the formation of nitrides leads to higher hardness.

The microstructure of the plasma gas nitrided Ti-8Al-1Mo-1V specimens using a power of 18 kW in the studied time intervals is homogeneous. As the power rises, leading the temperature on the surface of the treated samples beyond the β -transus, the microstructure changes to heterogeneous.

The surface hardness of the nitrided sample increases simultaneously with power and nitriding time. The increased surface hardness is due to the nitrogen diffusion and the newly formed phases – TiN and TiO₂ during the plasma gas nitriding process. Based on the measured microhardness values, the thickness of the nitrided layer is estimated to vary between 100 μ m and 350 μ m depending on the technological regime.

Acknowledgments

This work was supported by the Bulgarian National Science Fund within the M-ERA.NET Programme, Project “New generation copper-based coatings of improved antimicrobial resistance to pathogens”, No. KP-06-DO-02/1.

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