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INFLUENCE OF PVD COATINGS ON FATIGUE PROPERTIES OF HYBRID LAYERS IN HIGH TEMPERATURES

Key words

Hybrid layer, fatigue resistance, forging dies.

Abstract

Thermal fatigue is one of the most important mechanism of destroying tools operating in high temperatures, such as forging dies. Cyclical changes in the temperature gradient are a source of variable thermal stresses that cause the generation and propagation of thermal cracking in forging dies. Very effective operation of increasing the thermal fatigue resistance of forging dies is to shape the properties of the surface layer using surface engineering technology. One of the most perspective directions of the development of surface engineering is related to hybrid technologies. The best-known and widely used hybrid surface treatment technology is a combination plasma nitriding process with the PVD methods. The article presents the analysis of the impact of the different PVD coatings on the mechanism and intensity of the generation and propagation of cracks in the thermal fatigue hybrid layers “nitrided layer / PVD coating.”

Introduction

Thermal fatigue is one of the most important mechanism of destroying tools operating under large cyclic temperature changes, such as forging dies [1]. Cyclical changes in the temperature gradient between the surface and the tools' core are a source of variable thermal stresses that cause the generation and propagation of thermal cracking. Very effective operation of increasing the thermal fatigue resistance of forging dies is to shape the properties of the surface layer using surface engineering technology. One of the most perspective directions of the development of surface engineering is related to hybrid technologies [2], which best meet the expectation of the industry concerning the obtainment of the proper durability of forging dies. Hybrid technologies of surface treatment, being a combination of single processes in one multi-stage, continuous technological process, belong to the most advanced technologies in surface engineering. The best known and widely employed hybrid technology of surface treatment using the diffusion phenomenon is the combination of gas nitriding or ion nitriding followed by the deposition of hard, wear resistant coatings by means of PVD methods [3, 4]. A simultaneous occurrence of the two presented structure elements, i.e. the nitrided layer and PVD coating, results in their synergic interaction. The nitrided layer increases the surface hardness and substrate resistance to plastic deformation in the near-surface zone. This gives higher stiffness to the substrate-coating system, which protects the PVD coating from a loss of internal cohesion and adhesion to the substrate. The consistent PVD coating constitutes a barrier isolating the substrate nitrided surface and limiting the influence of external impacts on its destruction process. Due to special properties of the hybrid layer of the nitrided layer /PVD coating type, hybrid layers are more widely applied in industry, particularly due to the fact that they increase the durability of tools, including dies for aluminium pressure castings [5, 6], tools for cold plastic treatment [7], and hot forging dies [8, 9]. Examples of the practical application of hybrid layers of the nitrided layer/PVD coating type confirm their effectiveness in increasing the durability of tools utilised in very difficult working conditions. They clearly indicate the great importance of the correct maintenance, condition-adjusted selection of hybrid layer components, including the particularly thin PVD coating. The article presents the analysis of the impact of the different PVD coatings (CrN and AlCrTiN) on the mechanism and intensity of the generation and propagation of cracks in the thermal fatigue hybrid layers "nitrided layer / PVD coating."

1. Experimental details

1.1. Preparation of hybrid layers

Samples for investigations were made of hot working steel (EN X32CrMoV3.3) hardened by means of heat treatment (48-50 HRC) in a vacuum furnace (VPT 10.0 4035/36IQ/K Seco/Warwick), according to the requirements of the steel manufacturer – Bohler [10]. The investigated PN + PVD hybrid layers were created by means of the duplex surface treatment method in a two-stage continuous technological cycle, carried out at the same technological device: stage 1 – plasma nitriding, stage 2 – arc-evaporation of PVD coating. The plasma nitriding process was conducted by means of a special Optical Control System for the control of the intensity of plasma nitriding processes. The PVD coating deposition process was executed by means of the arc-evaporation method with the use of CDS-*Standard* device designed and produced by the Institute for Sustainable Technologies in Radom. In the second stage of duplex treatment, two different PVD coatings were deposited: CrN and AlCrTiN. The CrN coatings were made using pure Cr targets. The AlCrTiN coating was obtained using two different types of targets, including pure Ti and a compound target 70% Al + 30% Cr placed opposite each other. Parameters of the hybrid surface treatment technology are presented in Table 1.

Table 1. Parameters of the hybrid surface treatment technology

Stage	Stage name	Temperature T [°C]	U_{bias} [V]	Time t [min]	Pressure p [mbar]	Atmosphere
Stage 1	Heating	to 520		–	2.5	25%Ar+75%H_2
	Nitriding	520		420	4.3	15%N_2+85%H_2
	Cooling	–	–	60	–	–
	Ion etching	do 420	-950	1	< 10^{-4}	–
	Breake	–	-	1	< 10^{-4}	–
Stage 2	CrN	T_p = 400 T_k = 330	-200	120	3.5x10^{-2}	100%N_2
	AlCrTiN	T_p = 400 T_k = 330	-150	200	3.5x10^{-2}	100%N_2
	Cooling	<200	–	120	< 10^{-4}	–

1.2. Characterization of hybrid layers

The created composites underwent investigations aimed at defining their materials properties. After the deposition process, in order to determine the microstructure of the nitriding layer and PVD coating, respectively, microscope observations using a digital microscope (KEYENS VHX-1000E) and a scanning ion microscope (HITACHI FIB2100) were performed. Hardness measurements (HV0.5) in a function of the distance from the surface of the nitrided layer were conducted using a Vickers hardness tester FV7. The chemical composition of investigated PVD coatings was determined with the GDOES method with the use of Jobin Yvon JY10000RF Optical Spectrometer. Hardness and Young's modulus of particular PVD coatings were measured by means of the nano-indentation method, which used the Nano-Hardness Tester of CSM Instruments. Measurements were carried out with a Berkovich indenter in a single cycle using the following parameters: F = 10 mN, dF/dt = 20 mN/min. Based on the results, the hardness and Young's modulus were determined ("plasticity index" H^3/E^2), which is widely quoted as a valuable measure in determining the limit of elastic behaviour in a surface contact, which is clearly important for the avoidance of wear [11]. Coating adhesion investigations were carried out using the scratch-test method, which used a Revetest (CSM Instruments) equipped with a diamond Rockwell indenter. The indenter's load force was increased linearly in the range of 0–200 N, and the loading rate was 10N/min. Based on the changes in acoustic emission and friction force, three critical load forces were determined, corresponding to major coating damage failure: Fc1 – cohesion cracks, Fc2 – adhesion defects, and Fc3 – removed coating from all breadth of scratch.

1.3. Fatigue tests

For the fatigue tests of investigated hybrid layers, a die made of EN X32CrMoV3.3 hot working steel with a cylindrical shape and with a centrally placed pin in the form of a truncated cone [12] was selected. In order to assess the effect of different hybrid layers on fatigue properties, the forging dies with PN+CrN and PN+AlCrTiN hybrid layers were subject to maintenance testing and made appropriately 1, 100, 1000, and 2000 forgings. After the forging process, the samples were prepared according to the metallographic examination, whose aim was to determine the average density of cracks and the average length of cracks adequately for a different number of forging cycles.

2. Results

2.1. Characterization of hybrid layers

The hardness profiles of the plasma nitrided layer are presented in Fig. 1.

Nitrided layers are characterized by a similar surface hardness in the range of 890 – 920 HV0.5 and a comparable effective thickness at the level of 0.13 mm.

The chemical composition and microstructure of PVD coatings are presented in Fig. 2 and Fig. 3.

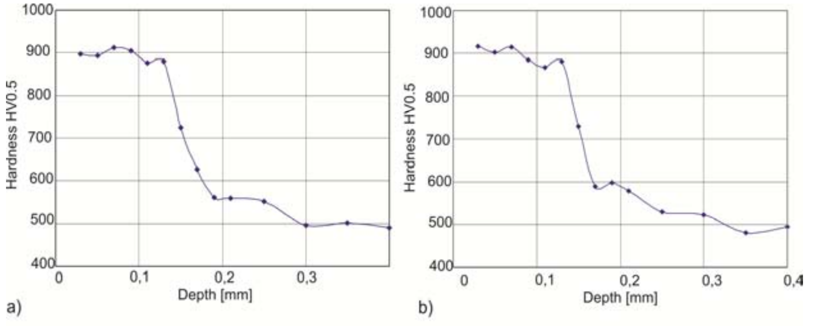

Fig. 1. Hardness profiles of investigated PN+CrN and PN+AlCrTiN hybrid layers

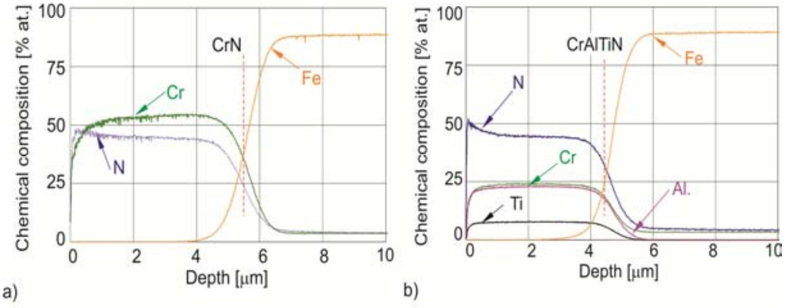

Fig. 2. GDOES depth profile of CrN and AlCrTiN coatings

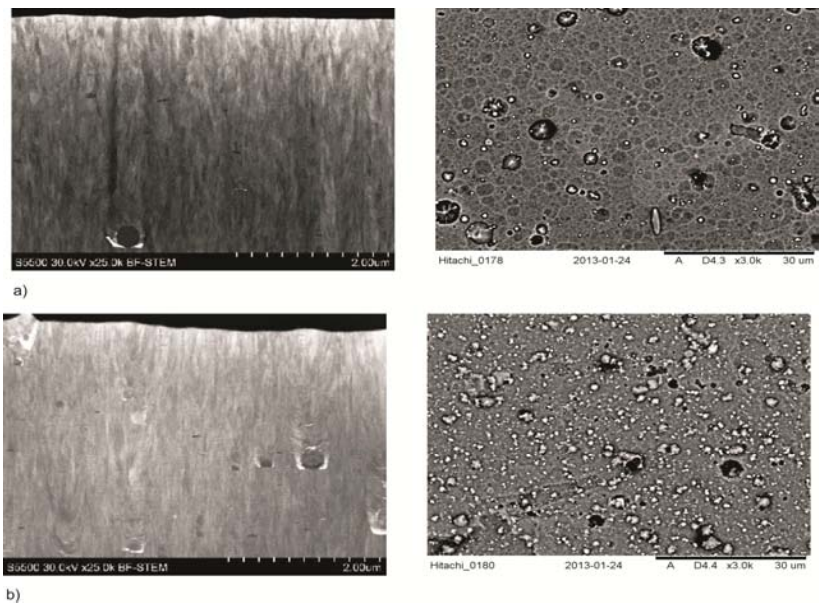

Fig. 3. Microstructure of CrN and AlCrTiN coatings

The examination of microstructures showed that the selected coatings are characterized by different droplet phase content. CrN coatings are characterized by lower rate of droplet phase. Both had the studied film microstructure of the transition zone model of Thornton (Zone T) [13].

The basic properties of the deposited PVD coatings are presented in Table 2.

Table 2. Properties of the deposited PVD coatings

Coating	Thickness *μm*	Hardness *GPa*	Young modulus *GPa*	H^3/E^2	Adhesion $F_{c1}/F_{c2}/F_{c3}$ *N*
CrN	4.5	21–23	260–300	≈0.13	46/60/103
AlCrTiN	4.0	28–30	320–350	≈0.20	–/83/110

Analysis of the results of the research material showed that the AlCrTiN coating is characterized by higher hardness and Young's modulus than the CrN coating. Adhesion test results showed that the AlCrTiN coating, despite the large amount of droplet phase, is characterized by better adhesion to the steel substrate than the CrN coating.

2.3. Fatigue tests

The results of the fatigue resistance of investigated hybrid layers PN+CrN and PN+AlCrTiN are shown in Figures 4, 5, and 6.

The analysis of changes in the average density of cracks and the average length of cracks, for different number of forging cycles, proved two different mechanisms of fatigue wear (Figs. 4 and 5).

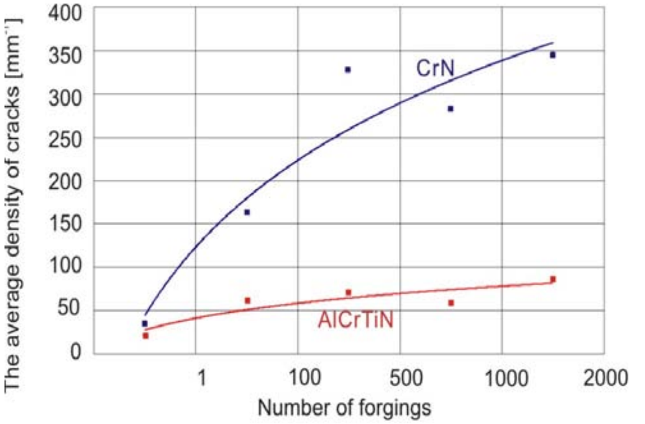

Fig. 4. The average density of crack formed as a result of fatigue tests executed for different forging amounts for the investigated PN+CrN and PN+AlCrTiN hybrid layers

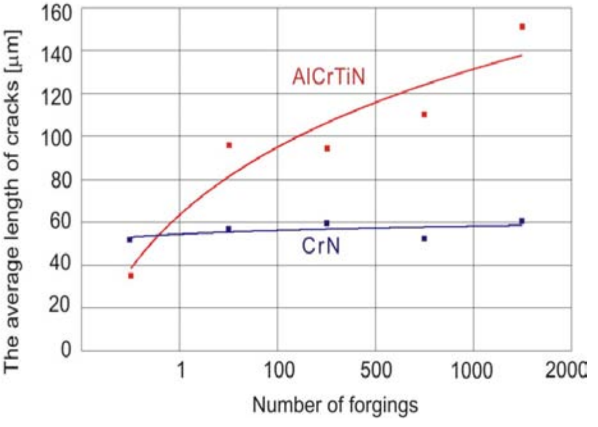

Fig. 5. The average length of crack formed as a result of fatigue tests executed for different forging amounts for the investigated PN+CrN and PN+AlCrTiN hybrid layers

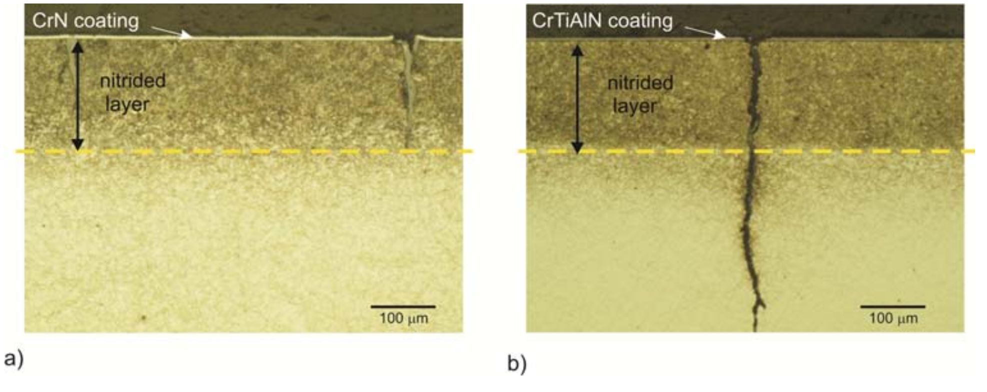

Fig. 6. The results of metallographic examination PN+CrN and PN+AlCrTiN after fatigue tests

The first one is characterised by the growing number of cracks, but the length of all cracks is shorter than the thickness of the nitrided layer (<60 µm), which was the case for the PN+CrN hybrid layer (Fig. 6a). The other fatigue wear mechanisms observed are characterised by a small number of cracks, but the length of all cracks is longer than the thickness of the nitrided layer (>120 µm), which was the case for the PN+AlCrTiN hybrid layer (Fig. 6b). The results of fatigue tests proved that the PN+CrN hybrid layer is characterised by better fatigue resistance than the PN+AlCrTiN layer. A large number of short cracks will cause the external loads to undergo greater dispersion. As a result, the energy focusing in a single burst will be less, and the propagation of cracks and new generation will be limited.

Conclusions

The obtained research results enable the formulation of the following conclusions:

1. During the fatigue resistance test of hybrid layers, two different mechanisms of fatigue wear were identified. The first one is characterised by the growing number of short cracks occurring for the PN+CrN hybrid layer, whereas the other one is characterized by a small number of long cracks occurring for the PN+AlCrTiN hybrid layer.
2. An increase of metal nitrides complexity in the coating causes the increase of the H3/E2 coating parameter, which is associated to the coating's resistance to plastic deformation, was observed. The disadvantageous effect of this change was the decrease of the fatigue resistance of PN+AlCrTiN in comparison to PN+CrN.
3. The properties of PVD coating, e.g. the complexity of metal nitrides, fulfil a very important role in the process of moulding the properties of hybrid layers PN+PVD.

Acknowledgements

Scientific work executed within the Strategic Programme "Innovative Systems of Technical Support for Sustainable Development of Economy" within Innovative Economy Operational Programme.

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Wpływ powłoki PVD na właściwości zmęczeniowe warstw hybrydowych w podwyższonych temperaturach

Słowa kluczowe

Warstwa hybrydowa, odporność na zmęczenie cieplne, matryce kuźnicze.

Streszczenie

Zmęczenie cieplne jest jednym z najbardziej istotnych mechanizmów niszczenia narzędzi pracujących w warunkach intensywnych, cyklicznie zmiennych obciążeń cieplnych, jak np. matryce kuźnicze. Cykliczne zmiany gradientu temperatury pomiędzy powierzchnią i rdzeniem narzędzia są źródłem zmiennych naprężeń cieplnych, które są przyczyną generowania i propagacji pęknięć w matrycy kuźniczej. Bardzo skutecznym działaniem zwiększającym odporności na zmęczenie cieplne matryc kuźniczych jest kształtowanie właściwości ich warstwy wierzchniej poprzez wykorzystanie nowoczesnych metod inżynierii

powierzchni. Jednym z najbardziej perspektywicznych kierunków rozwoju rozwiązań inżynierii powierzchni są technologie hybrydowe. Najlepiej dotąd poznaną technologią hybrydową jest technologia łącząca proces azotowania plazmowego oraz proces osadzania powłoki metodą PVD. W artykule przedstawiono analizę wpływu różnych powłok PVD na mechanizm oraz intensywność generowania i propagacji pęknięć w procesie zmęczenia cieplnego warstw hybrydowych typu „warstwa azotowana + powłoka PVD”.