Hot die forging with nitrided and thermally stabilized DLC coated tools

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Abstract. Hot forging dies are subjected to high loads, which can lead to early tool failures. Abrasive wear, plastic deformation and thermal softening of the surface layer can be counteracted in particular by a high surface hardness. Thermochemical diffusion treatments and coatings are established as wear protection measures. DLC coatings, which feature excellent frictional properties and high hardness, are commonly applied on cold forging tools. However, the low coating adhesion to steel and the thermal stability of the diamond bond limit the current range of application. In this study, DLC coatings are applied in metallic treatment atmospheres with the aim of increasing the diamond bond's temperature resistance. Furthermore, the influence of weak and intense nitriding to coating adhesion is investigated to reduce coating delamination. A pre-selection of modified DLC coatings for hot forging dies was carried out on the basis of hardness and scratch tests. The most promising tungsten DLC coating was tested in serial forging tests. Based on tool contour comparisons before and after forging, the potential as a wear protection measure for hot forging dies was determined. Tool wear was reduced by up to 29 % after 100 forging cycles with the tungsten DLC coating compared to the nitrided reference.

Introduction

Hot die forging is an economical process for the production of dynamically loaded high strength components in large quantities. About 15 % of the economic efficiency of forging processes is determined by tooling and setup costs [1]. An increased tool life can reduce these costs, enabling the forging industry to compete on an international market.

The primary limiting factor for the lifetime of hot forging dies is wear, which results from the high mechanical, thermal, tribological and chemical process-related loads. Abrasive wear describes the loss of material from the surface of a solid component caused by the contact and relative movement of an opposing object. In addition, these superimposed loads can cause plastic deformation and cracking [2]. The surface layer of the forging dies softens in contact with the hot blanks, which promotes abrasive wear and plastic deformation [3]. The use of cooling lubricants reduces the thermal tool load. However, the resulting cyclic temperature change leads to tensile and compressive stress changes in the tool surface layer, which can cause the formation of thermomechanical crack networks [4].

Common wear protection measures for hot forging dies aim to increase the hardness of the surface layer, to counteract abrasion, plastic deformation and thermal softening. Therefore, thermomechanical diffusion treatments such as nitriding are established processes. Diffusion of the nitrogen into the tool surface layer results in the formation of a nitrided zone. It consists of a compound layer and a diffusion zone. The compound layer is a complete conversion of the steel surface in iron nitrides. In the diffusion zone nitride precipitations are formed. Coatings can be applied to further increase hardness and thus wear resistance. Duplex treatments are commonly used, i.e. the coating is applied on nitrided surfaces. Nitriding counteracts early coating cracking, which is associated with softer surface layers below the coating (also known as “egg shall” effect) [5].

In cold forming processes, diamond-like carbon coatings are used for wear protection of forging dies [6]. The amorphous hydrogen-containing layers exhibit stochastically distributed networks of carbon and hydrogen. Both sp^2- and sp^3-hybridized forms of carbon are simultaneously present [7]. The first form describes the graphite bond and is known for its excellent friction properties. In sp^3-hybridized form (diamond bond), the carbon atoms are arranged as a tetrahedron, resulting in a high hardness of 1500 – 3500 HV [8]. A major disadvantage of DLC coatings is their low adhesion to steel, which leads to an early coating delamination. This is related to an inappropriate residual stress condition due to the application process [9]. Li et al investigated the influence of the sp^3/sp^2-ratio on DLC coating adhesion. They determined that an increased sp^3 content increases the hardness, but also reduces the toughness and thereby the adhesion of the coating [10]. Another promising approach to increase coating adhesion is prior plasma nitriding, which results in a less precipitous hardness gradient [11]. Due to the diamond bond’s low thermal stability, the field of applications of DLC coatings is limited, i.e. decomposition in air occurs at approximately 700°C and under loads earlier. According to Behrens et al, temperature resistance can be increased by metallic elements in the treatment atmosphere. This has been proven in particular for the element’s chromium and tungsten [12].

In this study, modified treatment atmospheres for DLC coatings containing the previously mentioned and other metallic elements are investigated with the aim of delaying or shifting the coating decomposition to higher temperatures. Furthermore, a suitable nitriding to increase the adhesion of the coating is to be determined. This is intended to enable the coatings to withstand the loads from hot die forging (up to 1200°C, currently up to 700°C).

Methods

First, the procedure for specimen and tool fabrication is explained. Furthermore, the surface layer modifications are described. Appropriate coating characterization methods are used to determine the most promising DLC coating which is applied to hot forging dies. The DLC coated tools and nitrided references are used in serial forging tests. Wear behavior is investigated by high-resolution images, contour comparisons and metallographic analysis.

Specimen and tool fabrication.

All specimens (ø35 mm x 4 mm) and tools were made of hot work tool steel AISI H11 and quenched and tempered to a hardness of 48 ± 2 HRC. Prior to nitriding, the surface was blasted with glass beads and cleaned.

Surface modification.

The surface modification includes nitriding and DLC coating of specimens and tools. A pre-selection of suitable DLC coating systems for hot forging dies is determined on the basis of microhardness tests and scratch tests.

Nitriding.

Two different nitriding processes were applied to the specimens (co. Rübig Anlagentechnik, type PN 100/150). Both nitriding treatments were carried out at a temperature of 520°C and for a duration of 16 h. The pulse-pause ratio was 100 µs (pulse) to 500 µs (pause). A pressure of 300 Pa was adjusted in the treatment chamber. As a significant difference, the nitrogen content in the treatment atmosphere was varied (10 % and 80 % N_2), the other part of the atmosphere was hydrogen.

DLC-Coating.

In total, three metal-containing DLC coating systems were applied to previously nitrided specimens (10 % and 80 % N_2) with the aim of increasing temperature resistance. The DLC coatings were applied on a PACVD coating system. Niobium (40 %) and tungsten (30 %) were used as atmosphere modifying elements (Nb40 and W30), due to the high potential already identified in previous studies [12]. In addition, a combined DLC coating system containing the elements niobium (20 %) and chromium (20 %) was investigated (Nb20Cr20).

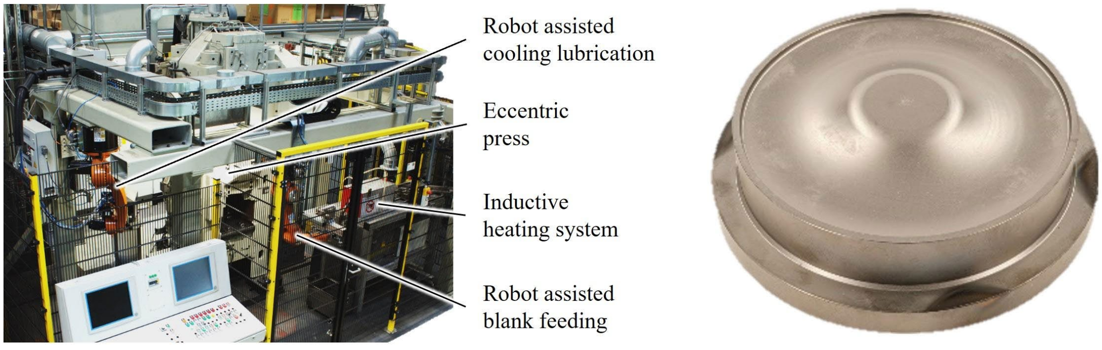
Layer characterization.

To determine the influence of the metallic elements in the treatment atmosphere on wear-reducing properties, such as high hardness, microhardness measurements according to Vickers were carried out. These were performed according to DIN EN ISO 6507 with a microhardness tester (co. Qness, type Q10A+). The hardness of the DLC coating was measured three times for each specimen and the standard deviation was determined. Furthermore, the Lc2 value was determined three times for each specimen after tempering the specimen at 600°C in scratch tests according to DIN EN ISO 20502 (co. Rtec Instruments, type SMT-5000), which describes the delamination of coatings.

Serial Forging Tests.

For characterization of the application behavior and qualification of DLC coated hot forging dies, serial forging tests were carried out on an eccentric press (co. Eumuco, type SP30d). A fully automated blank feeding system, forged part removal and spray cooling provide reproducible process conditions (Fig. 1, left). The blanks were made of the steel AISI 4140 with the dimensions ø30 x 40 mm. An inductive push-through heating system was used to heat the blanks. In one series of experiments, the blanks were heated to 1200°C and in another to 900°C. Between the forging cycles, a graphite-containing lubricant (co. Carl Bechem, type Berulit 906 HP) was applied in a ratio of 1:10 mixed with water. The forging dies were preheated to a temperature of 180°C.

In the serial forging tests, a highly tribological loaded forging die geometry was investigated, which is characterized by long material flow distances (Fig. 1, right). The forging dies were loaded up to 100 (900°C) and 1500 forging cycles (1200°C). Based on the layer characterization, the most promising coating DLC (W30) was applied to the previously quenched, tempered and nitrided (10 % and 80 % N_2) tool surfaces. In addition to the coated hot forging dies, quenched and tempered (unnitrided) as well as nitrided (10 % and 80 % N_2) reference tools were investigated.



Fully automated forging process

Primary tribological loaded forging die

Fig. 1. Fully automated serial forging process (left) and high tribological loaded hot forging die (right).

Wear analysis.

The performance and wear progress were characterized in dependence of the forging cycles. For this purpose, high-resolution images of the wear-critical central elevated area were obtained (cf. Fig. 4) using a 3D profilometer (co. Keyence, type VR- 3200). Based on these images, an initial wear analysis was carried out. Additional images were taken of non-critical wear areas, which were used to reference the tool contours before and after forging (cf. Fig. 5). The area between the profiles represents the planimetric wear which was determined according to DIN 50320. Six contour measurements were defined for each tool. Due to the focus depth, the area of analysis was limited to ±14 mm from the tool center. All specimens were metallographically prepared and etched with Nital to examine the microstructure and surface layer through light microscopy (co. Zeiss, type LEO 1530).

Results and Discussion

First, the characterization of the nitrided and DLC coated surface layers (microhardness measurements and scratch tests) is presented. Afterwards, the high-resolution images, contour comparisons and metallographic analyses of the forging dies are discussed.

Nitriding. Both determined hardness depth profiles for the nitriding processes 10 % and 80 % N_2 are shown in Fig. 2 (left). At a high nitrogen content of 80 %, a maximum hardness of approximately 1310 HV 0.005 was determined. In comparison, the hardness depth profile of the 10 % N_2 nitriding shows a maximum hardness of approximately 1170 HV 0.005. As a result of the increased nitrogen content, the precipitation behavior of nitrides is affected, leading to these differences. In addition, the diffusion of the nitrogen deeper into the material was enabled. This results in an approximately two times higher nitriding depth. A comparable hardness gradient can be seen for both nitriding processes, which can be explained by the identical treatment time and temperature. Furthermore, an increased nitrogen content results in additional diffusion processes, which leads to a homogeneous nitrogen distribution. Consequently, two nitriding processes with different characteristics are available for the subsequent application of the DLC coatings in order to investigate the influence on the coating adhesion.

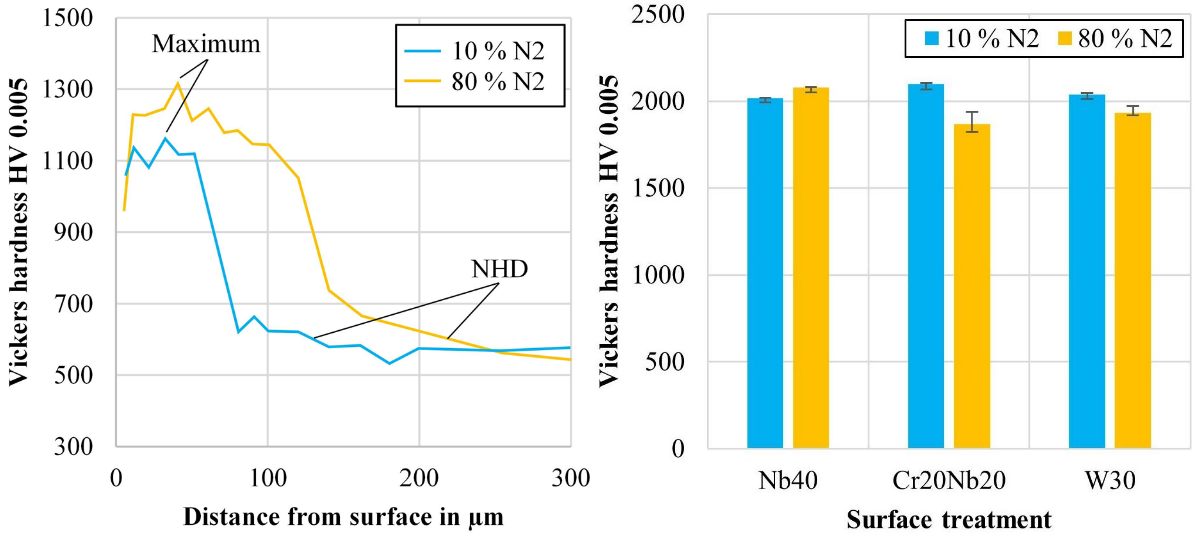


Fig. 2. Hardness depth profiles of the nitriding variants with a nitrogen content of 10 % and 80 % N_2 (left) and microhardness measurement on differently modified DLC coated specimens (right).

DLC Coating.

Cross-section microhardness tests were performed on the modified DLC coating systems Nb40, Cr20Nb20 and W30. The results are shown in Fig. 2 (right). It can be seen that the three DLC coating systems have a comparable hardness of approximately 2000 HV 0.005. Thus, for all variants, increased wear protection can be assumed compared to uncoated nitrided tools, if the DLC coatings withstand the thermal loads of hot die forging. Considering that the determined hardness is comparable to conventional DLC coatings [8], no negative influence of the modified treatment atmosphere can be observed.

Scratch tests were carried out to determine the most appropriate modified DLC coating system for hot die forging, and the results are shown in Fig. 3. The results show that the DLC coating system Nb40 is prone to early coating delamination at a temperature of 600°C (which approximately represents the maximum temperature of thermally loaded regions on hot forging dies). This behavior can be improved through the addition of the element chromium. The DLC coating system Cr20Nb20 features two to three times higher resistance to coating delamination. By combining DLC (W30) with 10 % N_2 nitriding, the highest coating resistance was observed. Therefore, DLC (W30) can be assumed to have the highest resistance to the loads associated with hot forging processes.

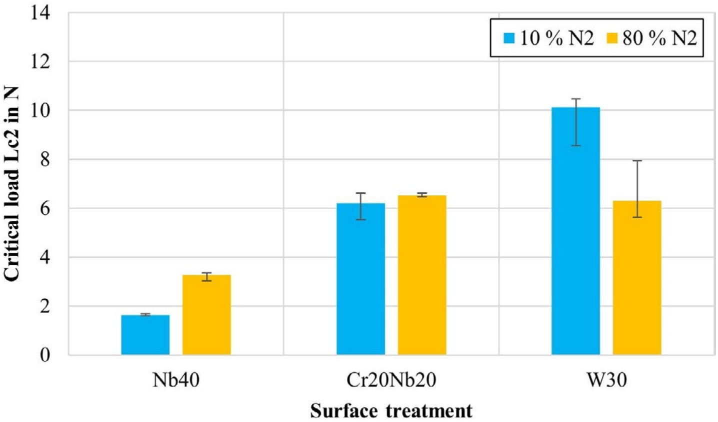
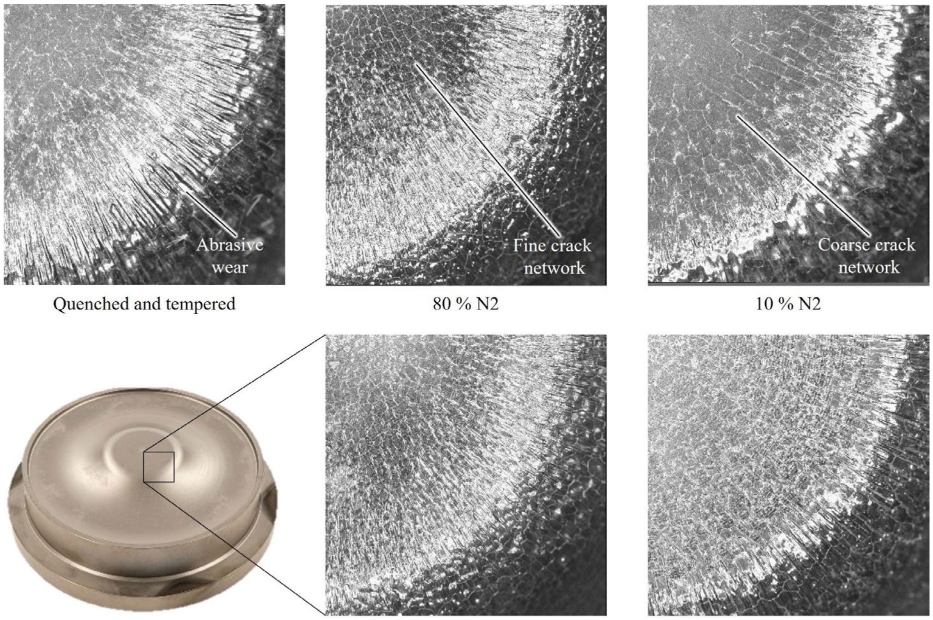


Fig. 3. Characterization of coating delamination at 600 °C of different metallic DLC coatings.

Wear analysis.

After the serial forging tests were carried out on the differently treated forging dies, high-resolution images were analyzed for the qualitative characterization of the wear behavior. The results after 1500 forging cycles are shown in Fig. 4. Maximum abrasive wear is present on the central elevated area radius of the quenched and tempered reference tool. In particular, the grooves in the direction of material flow are a characteristic feature of abrasion and a high tribological load. Due to the low hardness and significant thermal softening, this result was to be expected. Fine and coarse thermomechanical crack networks have formed in the middle of the central elevated area and near the radius. Intensively nitrided tools show an increased crack sensitivity compared to the 10 % N_2 variants. The DLC (W30) coated tools show finer crack networks than the nitrided reference tools and slightly reduced wear, therefore a coating removal can be assumed. This particularly affects the 10 % N_2 coated tool, since a lower hardness below the coating promotes cracking.



Quenched and tempered 80 % N2 10 % N2

Investigation area 80 % N2 + DLC (W30) 10 % N2 + DLC (W30)

Fig. 4. High-resolution images of the differently treated hot forging dies after 1500 forging cycles.

Contour Measurement.

The profile comparisons (Fig. 5, right) show a negative geometry deviation, which means abrasion is the main wear mechanism. Since no positive deviations are detectable, plastic deformation and adhesive wear either did not occur, or were superimposed by abrasion. Compared to the 10 % N_2 nitrided reference tool, a wear reduction of approximately 23 % was measured for the DLC coated forging die after 1500 forging cycles (Fig. 5, left). This correlation cannot be determined for the 80 % N_2 nitrided and DLC coated variant. A possible explanation is the almost two times higher layer adhesion, which can be observed from the results of the scratch tests (Fig. 3). After 100 forging cycles, the DLC coated forging dies exhibit less material removal than the nitrided reference dies. Abrasive wear was reduced by approximately 28 % with the DLC coated 80 % N_2 variant. In general, the lowest planimetric wear was observed after 1500 forging cycles for the uncoated 80 % N_2 nitrided tool.

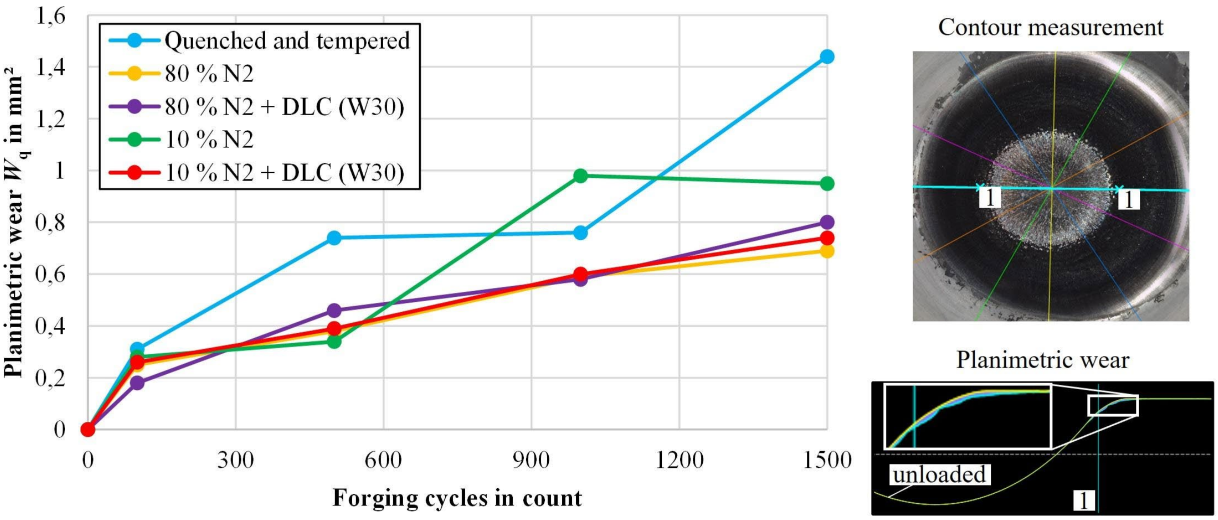
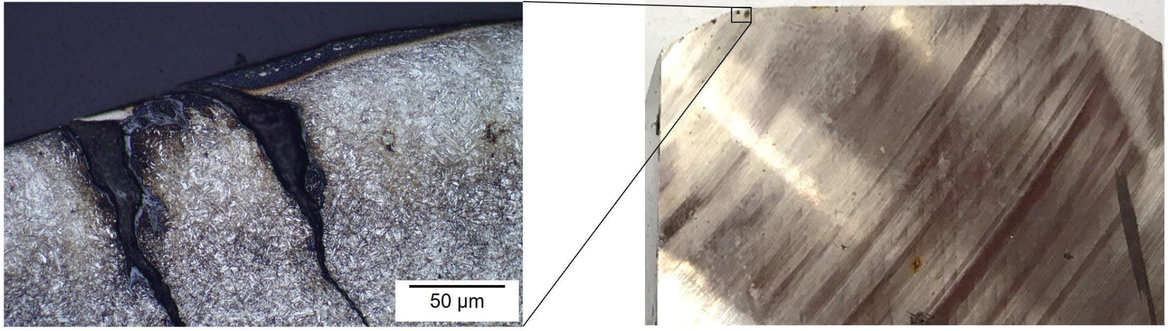


Fig. 5. Contour comparisons in dependence of the forging cycles of the differently treated forging dies.

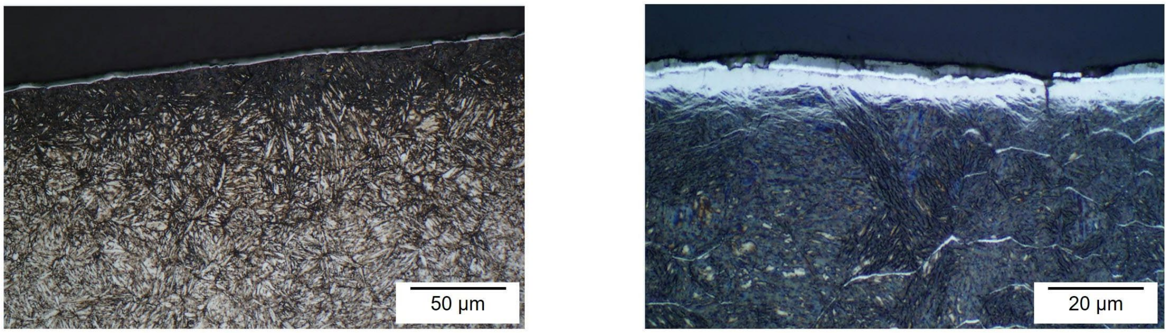
Metallography.

Metallographic cross-sections (Fig. 6) of the DLC coated tools were prepared and analyzed after reaching 100 forging cycles (900°C) or 1500 (1200°C). At a blank temperature of 1200°C and after 1500 forging cycles, significant cracks are detectable in the surface layer. Crack initiation was promoted by the hard surface as a result of nitriding. In the light gray zone, the surface layer has been thermally softened and the nitriding zone below is visible. The 10 % N_2 nitrided forging die with DLC coating no longer shows a coating after 1500 forging cycles at a blank temperature of 1200°C. Considering the contour comparisons (Fig. 5), the DLC coating seems to be present up to 500 forging cycles. Similarly, no DLC coating was observed on the 80 % N_2 nitrided tool after 1500 forging cycles. Additional serial forging tests at 900 °C were carried out with 100 forging cycles to confirm the increased thermal stability of the DLC (W30) coatings. Fig. 6 shows the results of the metallographic analysis and for both nitrided DLC coated variants (10 % and 80 % N_2) a thin white layer is visible, which is assumed to represent the DLC coating. The thick layer beneath it, is probably the compound layer. Similar to the serial forging tests at 1200°C, a maximum wear reduction of 29 % was found (80 % N_2).



1200 °C, 1500 forging cycles, 10 % N2 + DLC (W30)

Investigation area



900 °C, 100 forging cycles, 10 % N2 + DLC (W30)

900 °C, 100 forging cycles, 80 % N2 + DLC (W30)

Fig. 6. Metallographic analyses of the forging dies subjected to different loads and treatments.

Summary

To qualify metallic modified DLC coating systems as wear protection measures for hot forging tools, Nb40, Cr20Nb20 and W30 were applied to differently nitrided (10 % and 80 % N_2) specimens. Vickers hardness tests did not indicate any reduction of wear reducing properties such as high hardness through the modification. Scratch tests showed the highest resistance to coating delamination at a temperature of 600°C for the DLC (W30) coating (10 % N_2). Serial forging tests were carried out using a high tribological loaded forging die with blanks heated up to 900°C and 1200°C. After 1500 forging cycles, high-resolution images showed slightly lower wear and increased crack sensitivity for the DLC coated tools compared to the nitrided references. Based on contour comparisons, it was found that the DLC coated tool with 10 % N_2 nitriding had approximately 23 % less planimetric wear than the nitrided reference tool after 1500 forging cycles. A possible explanation is the increased layer adhesion, which was determined in the scratch tests. After 100 forging cycles and a blank temperature of 1200°C, a reduction in planimetric wear was observed for all DLC coated tools, which amounted to up to 28 %. Metallographic analysis showed no DLC coatings after 1500 forging cycles. Verification of higher temperature stability than conventional DLC coatings (700°C) was obtained through metallographic analysis of forging dies loaded with blanks heated to 900°C. Thin white layers are visible on these metallographic cross-sections, which are associated with an intact DLC coating.

Further destructive metallographic analyses are planned in the future to determine more precisely the layer degradation of the DLC coatings at a blank temperature of 1200°C. In addition, primary thermal loaded hot forging dies with modified DLC coatings are investigated to increase the range of applications.

Acknowledgment

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