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Improving Microstructure of AISI H13 Extruding Dies Using Ion Nitriding

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Abstract

A varied range of aluminum profile shapes are produced by hot extrusion and the die typically used is steel AISI H13. The quality and dimensions of the extruded pieces are determined by tribological and surface conditions of the die-bearing surface. Dies for hot extrusion require good metallurgical characteristics and chemical stability against hot aluminum. Experiments were conducted to evaluate the performance of specimens treated with conventional nitriding, and the ion nitriding process. Microstructural characterization, X-ray diffraction profiles, and wearing tests were carried out on the surface of H13 specimens. The ion nitriding process showed advantages over the conventional nitriding process, including productivity and tooling life.

Introduction

The extrusion of aluminum is a plastic deformation process in which a hot billet (usually round) is pressed at high pressure and temperature through a die of desired shape. Using this versatile process, it is possible to extrude profiles of almost any shape and length. Typical extrusion temperatures are around 550- 620 °C. At this temperature, the billet is forced through a die set by a ram hydraulic press causing extreme area reduction when the profile is formed [1]. Interaction between the surfaces of dies, and profile, during the extrusion process results in slide friction and adhesive wear which causes negative effects on the quality of extruded products, reducing die lifetimes and accelerating failure and replacement of the die set (Figure 1). Extrusion conditions demand that die materials have a high level of hardness, yield strength, creep resistance and toughness at elevated temperatures, as well as effective corrosion and wear resistance [2]. Metallurgical characteristics of AISI H13 make this tool-steel the most exclusively material used for hot work extrusion. Normally surface treatments are applied to H13 dies in order to increase the lifetime with the most popular surface hardening processes being carburizing and nitriding. Steel carburizing involves heat treatment of the metallic surface using a source of carbon, such as special atmosphere or a pack of carbonaceous material. Some typical hardening agents include carbon monoxide gas (CO) and sodium cyanide (NaCN). During carburizing, carbon is diffused into the surface layer of the workpiece at a high temperature, alloying and creating a high hardened surface. Similarly, during the nitriding process, nitrogen diffuses into the die surface to create a case hardened surface. The conventional process uses an ammonia atmosphere, or salt-bath containing nitrogen [3]. An innovation has used ion nitriding in which this plasma process produces more uniform cases, greater control over the process, and is cost effective. Another reason for the superior performance of ion nitrided cases is that the case is a monophase layer obtained without substantial modification of the bulk material properties [4].

Wearing of extrusion dies.

During the extrusion process, the die set is exposed to strong tribological loads through high contact (normal pressure) and sliding distances that generate adhesive and abrasive wear in the contact surfaces. It has been seen that when conventional nitriding is used, the die inlet surface is covered with a quasi-stable aluminum film. This film depends on factors such as speed, sliding, cycle time, temperature, and profile geometry. The film is renewable in a slow and discrete manner, permitting the die material to chemically react with it. Shear and tensile stresses developed during extrusion in combination with chemical reaction detach layer fragments and causing severe damage of nitride dies which predominately affects extrudate geometry. This causes severe wear of nitride dies in the form of wear craters and furrows on the bearing surface [5].

Experimental

Two sets of commercial steel AISI H13 of composition 0.39C, 0.45Mn, 1.05Si, 5.1Cr, 1.52Mo, 0.90V (wt. %) were used for experiments and tribological testing (3 witness samples per set). The conventional gas nitriding process was conducted in an industrial furnace equipped with automatic control of temperature and ammonia feeding system. Samples were preheated and maintained at 537 ± 5 °C for 10 hours. The internal furnace atmosphere consisted of a nitrogen and ammonia gas mixture. After the reaction and diffusion stage, a cooling fan was activated until room temperature was achieved which took approximately 3 hours. Samples for ion nitride were cleaned with acetone and placed in the Ionic chamber (vessel). The oxygen and other contaminants were evacuated and the vessel was filled with a reactive gas containing nitrogen. The electric power was turned on, and the gas was turned in an ionized state. The ion nitriding process was conducted in a commercial automated pulsed plasma nitriding system. The temperature and pressure conditions in the vessel were 530 °C, 5.5 mbar, respectively, with a controlled gas mixture of hydrogen and nitrogen flowing at 0.1 l/m. After the treatment, the samples were slowly cooled inside the vacuum chamber. The total ion nitride cycle took about 7.5 hours.

Tribological examination of samples was conducted using T-05 tester following the ASTM D2981, D 3704, G77 and D2714 standards. Optical microscopy, micro-hardness indentations on the deposited layer, and o SEM analysis were carried out on the samples focusing on nitrogen content added for each process.

Results and discussion

As seen in figure 2, the cross section of gas nitride surfaces showed a non-uniform case layer. The visual depth of the nitride layer as estimated from the optical micrograph was about 70-100 µm. The micro-hardness profile at the surface decreased in depth direction, the maximum hardness value (1099 HV) was located at the surface, and decay was across the nitride layer with values around 880-900 HV at the bottom of the case layer. Near the surface, the nitrogen content was maximum. This amount of released nitrogen created a monophasic layer with high hardness values [7].

The cross section of the ion-nitride sample is shown in figure 3. The main difference was that glow discharge formed a more uniform case, and from the optical micrograph the nitride layer was estimated at about 100 µm. The maximum hardness value was 1181 HV at the top surface, and 1115 HV in the bottom. Both processes developed a thin layer of Fe-nitride known as "white layer". Figures 4 and 5 show the results of linear XRD analysis. In these images, we can see that when surface treatments, such as gas and ion nitriding, are applied to steel AISI H13, some alloy elements precipitates (such as chromium, vanadium and molybdenum nitrides) contribute to the surface hardening of the base material [8].

Line-scan and elemental mapping analysis gave a semi-quantitative nitrogen concentration and measurements for each process are depicted in figure 6 as a box-plot graph. During the ion-nitriding process, the nitriding reaction occurred at surface and sub-surface, and diffused nitrogen into steel surface was combined with alloying elements to form several fine nitrides. According to literature the microstructure of the case layer developed by ion-nitriding processes near 540 °C consisted of only γ-Fe_4N phase. This monophase layer has high hardness and toughness, and supports high dynamics loads and abrasive wear [9,10].

Figures 7 and 8 show results of tribological testing (wearing and friction coefficient). These tests confirmed that ion nitriding has advantages over conventional gas nitriding [11]. In order to verify the results obtained in nitrided witness samples, two dies were taken and evaluated in an extrusion presss under the same production conditions (speed, alloy and temperature). Dies used as confirmatory trials were nitrided simultaneously with the samples. Figure 9 shows the favorable effect on productivity, and improvements in the service life if ion nitriding is selected.

Conclusions

- Ion nitriding is an efficient method for surface hardening of aluminum extrusion dies.
- The case formed by ion nitriding is more uniform than the conventional nitriding process. Visual depth of the ion nitrided was about 100 µm with the highest hardness value.
- Under production conditions, ion nitrided dies show productivity advantages and longer life than conventional gas nitriding process.

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Figures

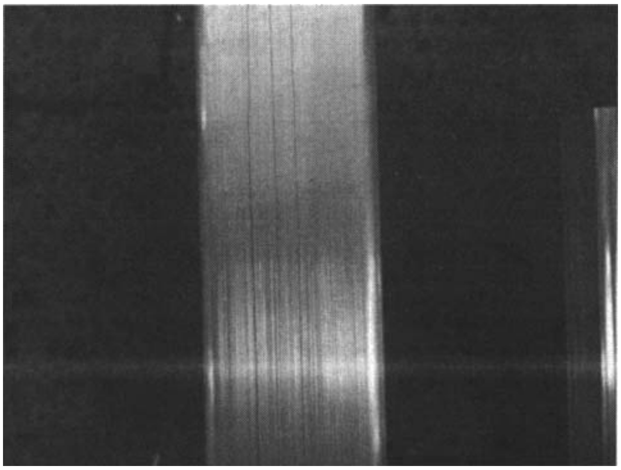

Figure 1Extrusion surface defect caused by low quality die condition.

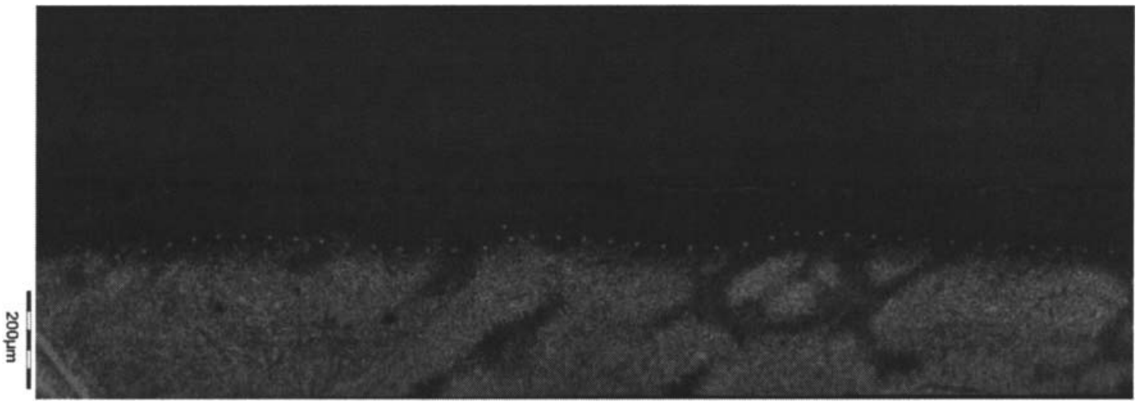

Figure 2 Optical microscopy of gas nitride microstructure showing a compound layer with variable thickness.

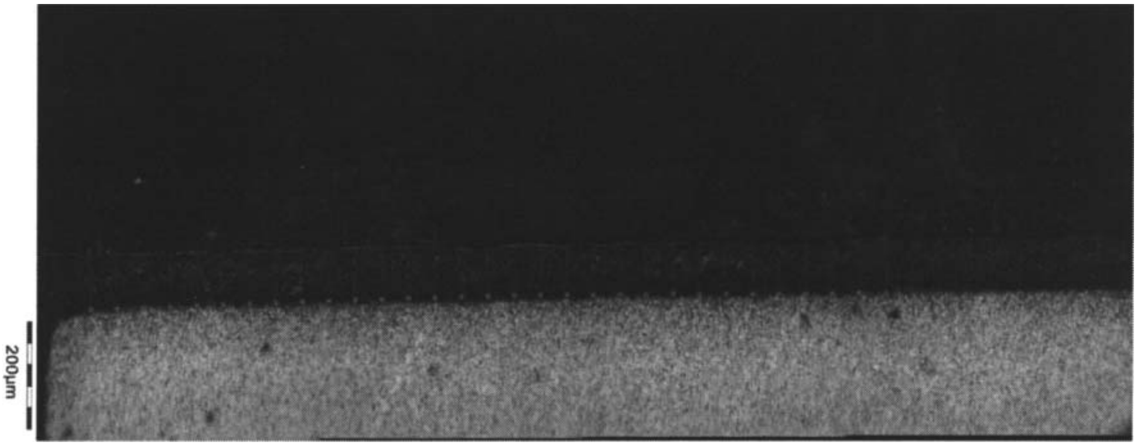

Figure 3 Optical microscopy of ion-nitride microstructure showing a uniform case layer.

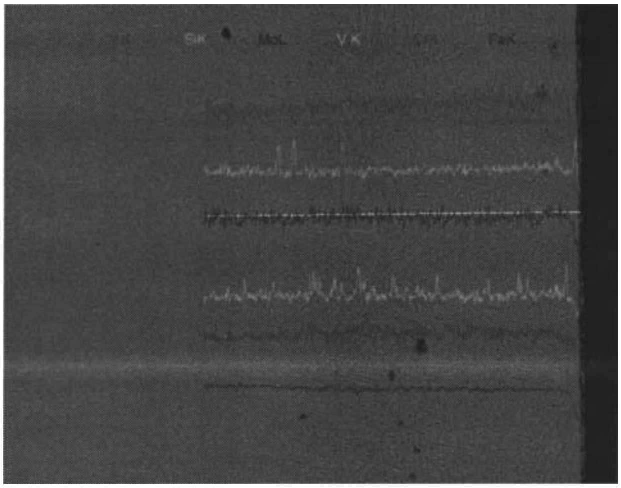

Figure 4. X ray diffraction patterns of gas nitride surface (linescan).

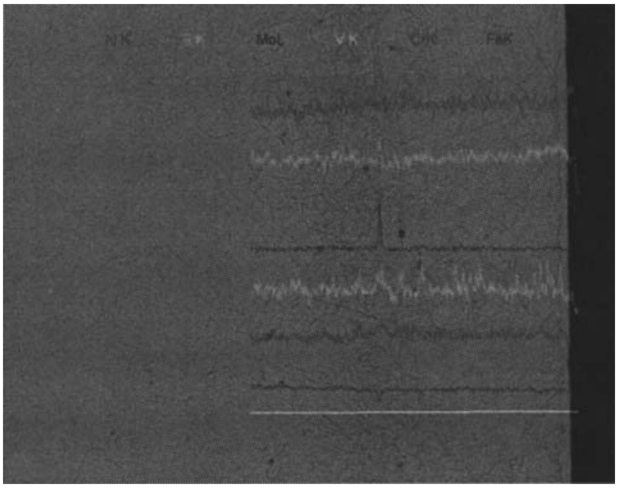

Figure 5. X ray diffraction patterns of gas ion-nitride surface (linescan).

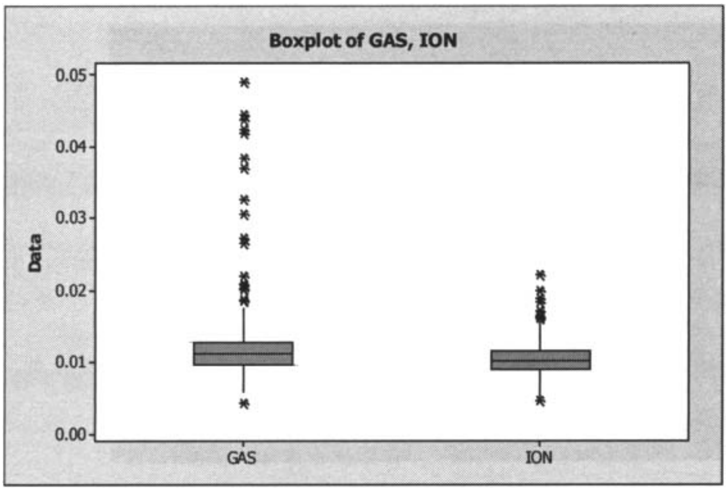

Figure 6 Box-plot showing semi-quantitative nitrogen concentrations in nitride layer.

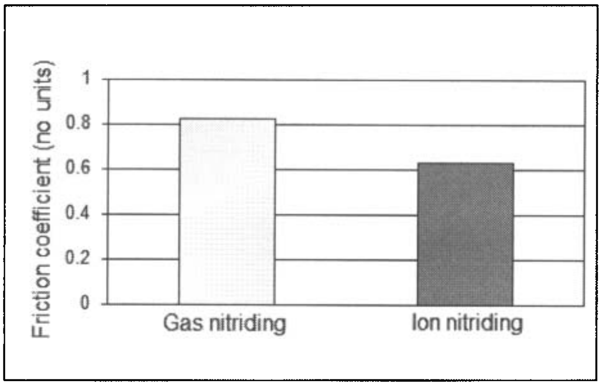

Figure 7 Layer material removed for different nitriding processes.

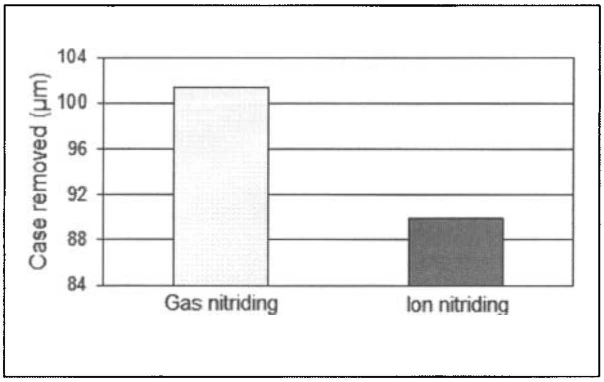

Figure 8 Average peak of friction coefficient for nitriding processes.

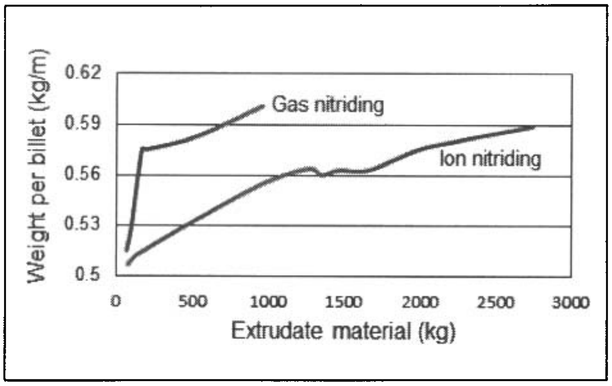

Figure 9 Weight developed per unit length of billets during production trials.