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Reducing the Surface Degradation of Aluminum Extrusion Dies During Preheating

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Aluminum extrusion dies are usually made from H13 steel that is ferritically nitrocarburized to minimize wear and pick-up. Before being placed in the extrusion press, the dies are preheated to minimize thermal shock at the start of the extrusion cycle. During the preheating time, the nitrocarburized layer oxidizes. Some of this layer can break away during extrusion leaving marks on the product. Although inerting the preheat furnaces with nitrogen has been found to reduce the oxidation, it does not solve the problem completely. Experiments have shown that a small addition of ammonia to the preheating protective atmosphere could eliminate oxidation and prevent nitrogen loss from the surface nitride layer.

Keywords aluminum, extrusion, heat treating, surface engineering, tool steels

1. Introduction

Aluminum extrusion dies are usually made from H13 steel. After standard hardening and form cutting, the dies are tested on the extrusion press and final adjustments made. Residual aluminum is removed in a caustic bath and the die is cleaned by grit blasting. The final process before use is a ferritic nitrocarburization process to minimize both wear and pick-up (Ref 1). The ferritic nitrocarburizing process is described in detail elsewhere (Ref 2). Basically, the steel is exposed to an atmosphere of approximately 50% nitrogen and 50% ammonia with a small oxidant addition (typically 3% carbon dioxide) at around 550 °C for several hours. In this process, a surface layer with a thickness of 5 to 20 μm is formed, consisting mainly of ε- and/or γ′-phase iron carbonitride, and a back-up diffusion layer about 0.05 to 0.3 mm thick in which the nitrogen is essentially in solution. Some typical nitrocarburized extrusion dies are shown in Fig. 1.

During extrusion, the aluminum billet is at a temperature between 450 and 520 °C. The extrusion die itself reaches about 600 °C. To avoid excessively large temperature gradients during the initial phase of the extrusion process, which might lead to die failure, each die is preheated to between 450 and 520 °C in a small furnace. This is usually carried out in air.

The dies may remain in these furnaces for 12 h before use if a large number of different tools are utilized in an extrusion plant but a typical time is around 4 h. During this time, the epsilon iron carbonitride surface layer oxidizes, forming a typical black surface. If this layer becomes too thick some may break away during extrusion, marking the surface of the extrusion (Ref 3). It is therefore desirable to avoid oxidation.

As well as producing a friable surface, the long preheating period has a negative influence on the diffusion layer of the nitrocarburized die. The nitrogen present in the supporting diffusion zone will start to diffuse again. Some of this nitrogen penetrates deeper into the tool and some is lost from the surface of the die. Consequently, the properties of the die will be changed and its resistance to wear decreases.

2. Experimental and Results

Initially, it was considered that inerting the preheating furnaces with nitrogen would be sufficient to eliminate or at least reduce oxidation. Unfortunately, the preheating furnace at the Belgian company where the trials took place was not designed for the atmosphere used. In particular, the seals on the circulation fan shafts were not able to stop them drawing in air. Even with a high flow of nitrogen it was impossible to achieve a low oxygen level and oxidation of the surface layer was almost unaffected.

XRD studies were carried out to determine the effect of oxygen on the nitrocarburized layer. In more controlled laboratory conditions it was shown that when the oxygen was reduced to a very low level, the oxidation rate could be reduced from 0.5 to 0.3 μm/h for a typical preheating temperature. However, it was not possible to eliminate oxidation completely, and even under nitrogen, the layer was completely destroyed after 30 h. That work is summarized in Table 1 and some typical XRD analysis data are shown in Fig. 2.

The objective of further work was therefore to overcome the problems described above and to find a better way to maintain the hardness of the carbonitride surface layer of the die. An active additive to the nitrogen was needed to prevent outward diffusion of nitrogen atoms, so maintaining the concentration of nitrogen in the surface layer. The active additive chosen was ammonia, which decomposes at the steel surface into nitrogen

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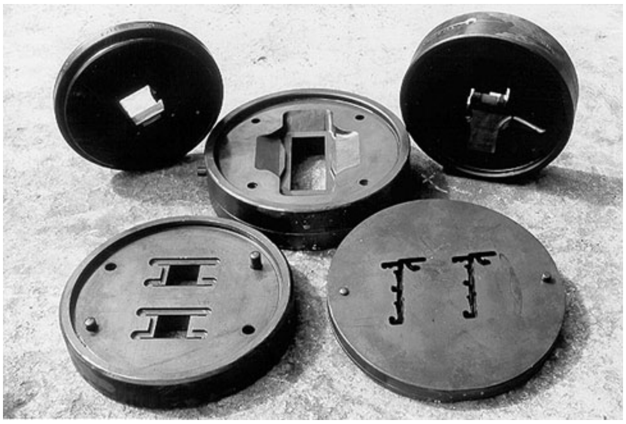

Fig. 1 Extrusion dies after NITROFLEX™ ferritic nitrocarburizing

Table 1 The effects of oxidation in various conditions

Temperature, °C	Atmosphere	Main comments
450	Air	After 4 h, the sample had an oxidized surface with some ε-nitride and γ′-nitride left in the layer penetrated by the x-ray. The samples that had spent 16/32 h in the furnace had only traces of γ′-nitride left.
450	Nitrogen in a laboratory furnace	After 4 h, the nitrided layer had oxidized, but not to the same extent as the sample that spent 4 h in the air furnace. Even after 16 h, a substantial part of the nitrided layer was left. After 30 h, the nitrided layer was almost totally oxidized. ε-Nitride degenerated faster than γ′-nitride, or possibly ε-nitride transformed to γ′-nitride.
490	Industrial furnace nitrogen inerted	Comparing these samples with the sample that spent 4 h in the laboratory furnace with protective gas, the degeneration for all these samples was almost the same.

atoms and hydrogen molecules. The nascent nitrogen diffuses into the tool steel and the hydrogen helps to prevent oxidation. By choosing a correct quantity of ammonia, it should be possible to maintain an essentially constant nitrogen level in the surface layer.

Samples of hardened and tempered H13 were gas nitrided using a typical industrial cycle at 530 °C for 6 h. The GDOES nitrogen profile, hardness profile, and microstructure after a typical ferritic nitrocarburizing treatment on H13 are shown in Fig. 3 to 5.

The samples were then kept at 450 °C for 30 h in atmospheres of air, nitrogen, or nitrogen/1% ammonia in a laboratory furnace. GDOES profiles were then performed with the results for the nitrogen profile shown in Fig. 6, the hardness profile in Fig. 7, and the XRD analysis in Fig. 8. The profile for the 1% ammonia treatment is obviously superior to the air and nitrogen treatments and has a slightly higher surface nitrogen than that for the original nitrocarburizing treatment. XRD analysis showed that there was an increase in γ′ in the nitride layer.

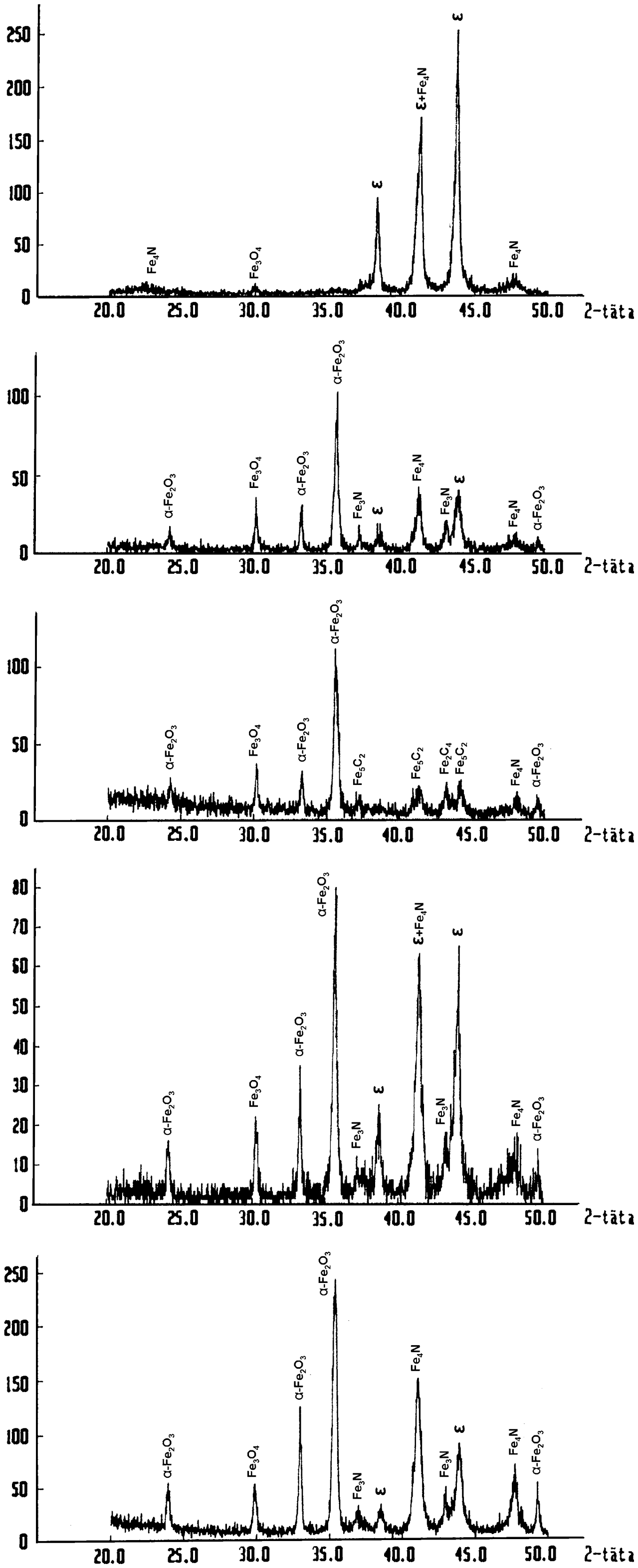

Fig. 2 The XRD analysis data for (a) a nitrocarburized die, (b) after 4 h at 450 °C in air, (c) after 16 h at 450 °C in air, (d) after 4 h at 450 °C in nitrogen, and (e) after 16 h at 450 °C in nitrogen

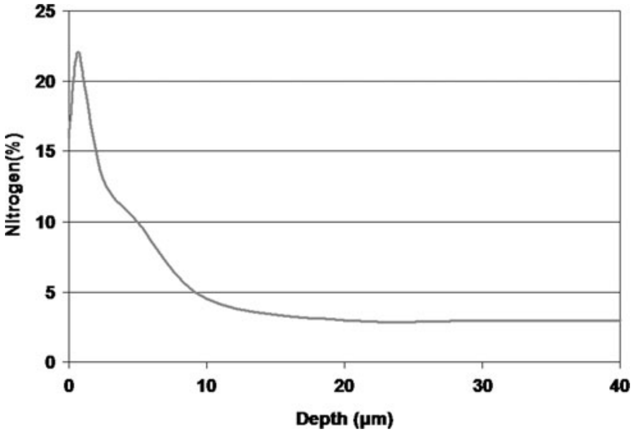

Fig. 3 The GDOES nitrogen profile for ferritic nitrocarburized H13

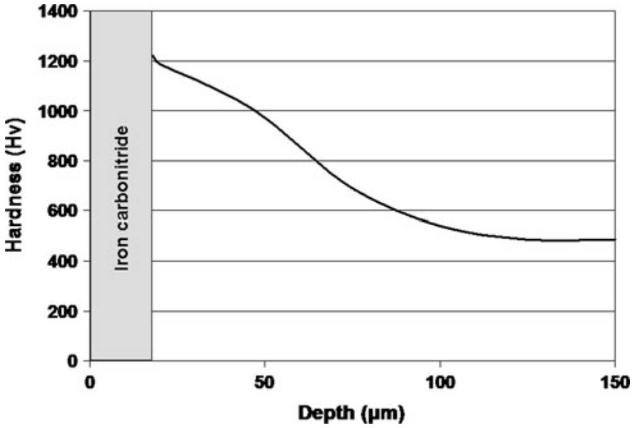

Fig. 4 The hardness profile for NITROFLEX™-treated H13

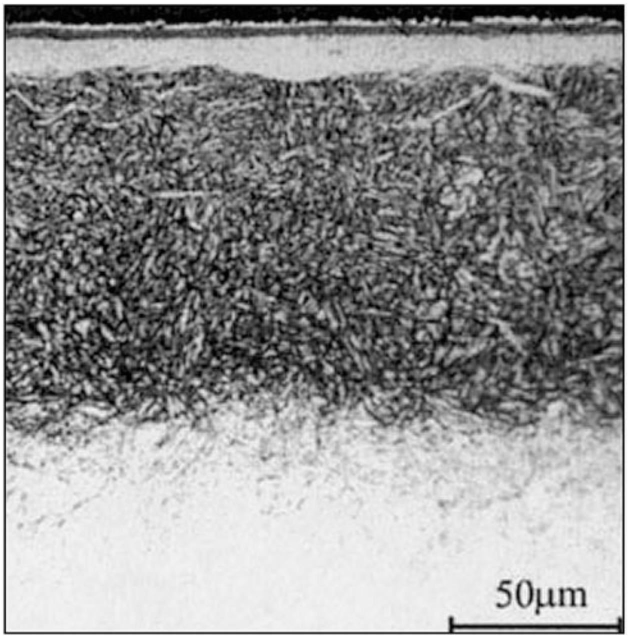

Fig. 5 The microstructure of a typical ferritically nitrocarburized H13

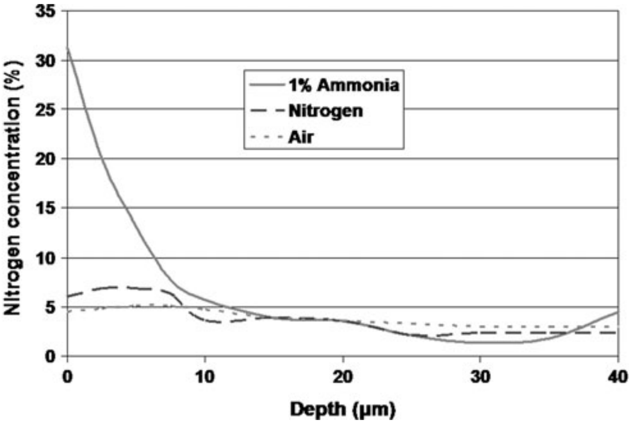

Fig. 6 GDOES nitrogen profiles for nitrocarburized H13 exposed to various atmospheres at 450 °C for 30 h

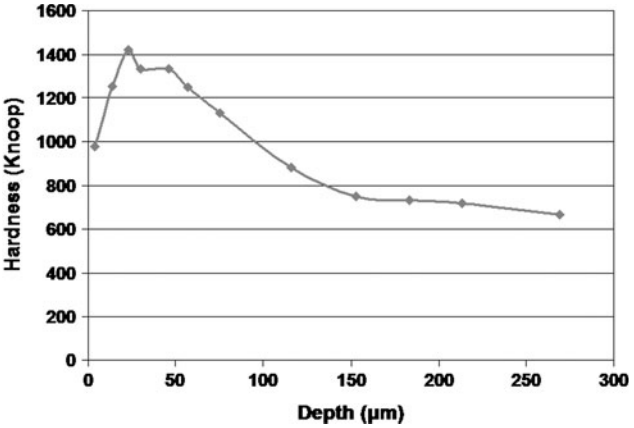

Fig. 7 The hardness profile of nitrocarburized H13 after 30 h at 450 °C in 1% ammonia

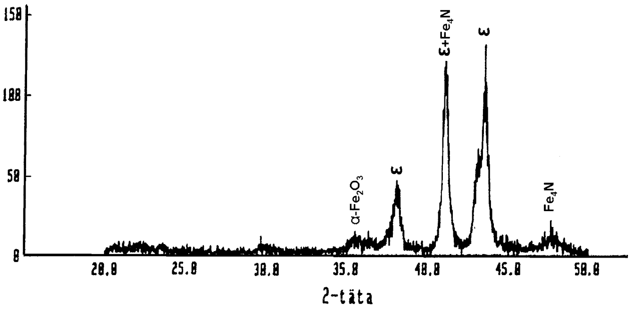

Fig. 8 The XRD analysis after 30 h in nitrogen/1% ammonia

3. Discussion

The nitrogen profile of the nitrocarburized layer initially formed on H13 in this study is almost exactly the same as that found by King et al. (Ref 4).

Lee has shown that after nitrocarburizing, the growth rate of the oxidation layer increased with increasing treatment temperature and time. The oxide layer is easily spalled from the compound layer either for an oxidation temperature above 450 °C or for an oxidation time longer than 2 h at a temperature of 400 °C. It was confirmed that the ratio of Fe_2O_3 to Fe_3O_4 increased rapidly as the oxidation temperature increased (Ref 5, 6). The rate of oxidation was broadly in line with the findings of Jutte et al. (Ref 7).

Hundred percent ε-nitride is known to give the best wear (Ref 8). However, it is also probable that the presence of some nitride rather than oxide on the surface will improve wear. Thus, the 1% ammonia treatment should improve wear, but perhaps not optimally. A slightly lower concentration might give a better result.

4. Conclusions

Using nitrogen alone is of little benefit in protecting the nitrocarburized layer from oxidation in the furnace used to reheat and store aluminum extrusion dies prior to use. The addition of a small quantity of ammonia to the nitrogen is effective in preventing oxidation and may even improve the layer.

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