Investigation of the properties of the material zone below alloy surfaces prepared by electrolytic belt grinding or electrochemical machining

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The properties of the material zone below surfaces prepared by electrolytic belt grinding or electrochemical machining of two superalloys were investigated by microhardness measurements, thermal probe traverses, microprobe scans and scanning microscopy. The shape of the microhardness profile was found to strongly depend upon the location of the measurements. A layer with a uniform hardness decrease away from the surface, was not found to exist. Either the defect structure in the region of the measurement or a generalized Rebinder effect may be responsible for the microhardness profile.

1. Introduction

The hardness in a thin zone below surfaces which were prepared by different non-conventional techniques like electrochemical machining (ECM), electrical discharge machining (EDM) and electrolytic belt grinding on superalloys was compared [1] recently. It was reported there that the hardness of electrochemically machined specimens at a depth of about 2.54×10^{-3} cm below the surface was smaller than that of the bulk. The hardness increased continuously with the distance from the surface and tended towards the bulk value, at about 0.013 cm for Inconel 718 alloy for instance. A similar behaviour was found [2] on electrochemically machined specimens of Titanium 6A1-4V, Inconel 718 alloy, 1018 Steel, 4340 Steel, 410 Steel, 302 Stainless Steel, Grade 250 Maraging Steel, Molybdenum Alloy T2M, Unalloyed Tungsten, Cast In 100, Cast Rene 41 and Waspaloy alloy. In general, the extent of the surface softening depended upon the machining conditions (gentle or abrasive) and was not always reproducible for each specimen of the same material.

The causes of the hardness decrease in the thin zone between the surface and the bulk are not known at present. The phenomenon appears to be similar to the so-called Rebinder effect [3] in which changes in mechanical properties result from surface tension changes induced by the adsorption of surface-active species. It is the purpose of this communication to report and discuss new results concerning the surface softening. An electrolytically belt-ground specimen of Hastelloy X alloy and an electrochemically machined specimen of Inconel 718 alloy were studied*. The electrolyte was about 2M NaCl (1 lb/gallon), used at 40°C in both operations. Machining was done at about 1000 A/in² at 20 V. The typical surface finish of the flat electrochemically machined surface is 20 to 24 microinches arithmetic average.

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2. Experimental

The Hastelloy X alloy specimen was cut into three pieces of about equal lengths. Crosssections perpendicular to the belt-ground surface were prepared by standard metallurgical procedures for each of these pieces (compare Fig. 1). The cross-section of the first piece in a



Fig. 1. Schematic drawing of the cross-section of the beltground specimen of Hastelloy X with regions of measurements.

suitable mount was used for microhardness tests, and that of the second piece for traverses by a thermal electric probe. The cross-section of the third piece in a conducting mount was investigated by microprobe scans. This specimen was removed from the mount and repolished before photos were taken by the scanning microscope. All the measurements were made in the indicated direction on that part of the cross-section where the curvature of the ground surface is relatively small (see Fig. 1).

The Inconel 718 alloy specimen had been electrochemically machined on two sides in such a way that two flat surfaces resulted. The cross-section perpendicular to the electrochemically machined surface is shown in Fig. 2



Fig. 2. Schematic drawing of the cross-section of the electrochemically machined specimen of Inconel 718 with regions of measurements.

with the locations and the directions of microhardness measurements. Only hardness determinations were made on the Inconel 718 sample.

The Vickers microhardness was determined at a load of 50 g, a loading rate of 0.05 mm s^{-1} and a 10 s duration of load. The indenter was of the DPH type. The measurements were attempted

along straight lines indicated by the arrows in Fig. 1 and Fig. 2. However, a slight shift to the side was sometimes necessary to avoid making an indentation at a visible defect. The spacing of the indentations was made smaller in the direct vicinity of the surface and larger at distances above 5×10^{-3} cm. An assembly for the measurement of the thermal EMF of the junction between the small tip (diameter about 20µm) of a thin tungsten wire and the alloy was constructed according to the details given in [4]. The alloy in the mount was kept at a constant temperature of about 60°C in a bath. The tungsten wire was at room temperature. The measurements were made in an apparatus for microhardness measurements in which the indenter with the diamond had been replaced by the tungsten wire probe. Since the thermal EMF was very small, the first experiment consisted of following the thermal EMF at a constant distance from the surface as a function of time during the heating up of the bath. Simultaneously the EMF of a thermocouple touching the specimen inside the mount was recorded. The comparison of the two measurements demonstrated that the same temperature change was reflected by the two EMF's. Thus the possibility that another voltage, other than the thermal EMF of the junction between the tip of the tungsten wire and the alloy, was measured, could be eliminated.

3. Results

3.1 Belt-ground specimen of Hastelloy X alloy.

In the first determination of the microhardness as a function of distance from the machined surface, two readings were taken at the same distance by shifting the specimen in the direction perpendicular to the direction of the measurement by about twice the diameter of the indenter. The magnitude of the scattering of the experimental values of the microhardness is indicated in the respective plot of Fig. 3. The scattering is relatively large. However, smooth curves may be constructed as hardness profiles by rough averaging. After repolishing the cross-section, a second run was made. The resulting hardness profile is shown in Fig. 3. For comparison, the microhardness was also determined as a function



Fig 3. Plots of microhardness versus distance from edge. \bigcirc , \triangle : belt ground surface \Box mechanically cut surface.

of distance from the mechanically cut surface (see Fig. 3). All the microhardness profiles were constructed under the reasonable assumption that at least two experimental points are required for the establishment of the general trend. The traverses with the thermal probe on the second sample of Hastelloy X alloy did not yield an identifiable change of the EMF with distance. The thermal EMF $(90 \pm 10\mu V)$ was practically independent of distance from the ground surface in the same region where the microhardness on the first sample of Hastelloy X alloy displayed a large variation.

Linear scans were made by microprobe over a distance of 80 μ m from the ground surface of the third Hastelloy X alloy sample with counts at 2μ m steps to look for possible changes of the concentration of the main constituents of the alloy: C, Cr, Co, Fe, Mg, Mo, P, Si, S, W, and Ni. The scans were located within the same area as the microhardness measurements on the first Hastelloy X alloy sample. Significant changes of the concentration of any of the above elements were not detected. It should be noted here that the sensitivity for the carbon detection was less than that for the other elements in question.

The rugged edge and part of the same region as that for the microhardness measurements on the first sample are shown for the third sample of Hastelloy X alloy at $1000 \times$ in Fig. 4. The picture was taken by the scanning microscope. As do other pictures at larger magnifications, Fig. 4 reveals the presence of numerous embedded



Fig 4. Picture of the cross-section close to the ground surface of Hastelloy X taken at a magnification of $1000 \times$ by the scanning microscope.

particles with average diameters between 0.2μ m and 0.5μ m. The composition of these particles is not known with certainty. Probably they are carbide particles. Pictures were also taken at distances of approximately 50μ m, 150μ m, and 250μ m from the surface, starting in the area marked by two arrows on the margins of Fig. 4. These pictures and visual observation of many more areas did not reveal large changes in the surface topography.

3.2. Electrochemically machined specimen

The microhardness is plotted as a function of distance from the upper and lower electrochemically machined surface in Fig. 5 and Fig. 6 respectively. The location and direction of the measurements are indicated by arrows in Fig. 2. The previous assumptions for the drawing of the microhardness profiles were employed.

After repolishing the sample, the results in Fig. 7 were obtained in positions D and E of the lower electrochemically machined surface. The microhardness measurements at position E were made by a Knoop indenter in order to check if a



Fig. 5. Microhardness as a function of distance from upper electrochemically machined surface.

o: at location A

 \triangle : at location B

□: at location C



Fig 6. Microhardness as a function of distance from lower electrochemically machined surface.

•: at location D

▲: at location E

■: at location F

surface softening is also observable with another type of indenter and a larger load. The respective microhardness profile in Fig. 7 demonstrates that this is the case.



Fig 7. Microhardness as a function of distance from lower electrochemically machined surface. ○: location D, Vicker □: location E, Knoop

4. Discussion

The results in Fig. 3 reveal that the microhardness of the mechanically cut surface is larger than that of the bulk of the first sample of Hastelloy X alloy. It passes through a wide minimum before it approaches an average bulk value of 250 DPH. In spite of the relatively large scattering it can be stated that the microhardness close to the belt-ground surface is smaller than the average bulk value. It increases subsequently to a maximum value of about 275 DPH, passes through a shallow minimum and tends towards the bulk value.

The thermal probe traverses which were made on the second sample in the same region as for the microhardness measurements on the first piece of Hastelloy X alloy do not give any evidence for composition changes of the superalloy in the studied region. Nor do the microprobe scans on the third sample and the scanning microscopy.

The microhardness measurements on the Inconel 718 alloy specimen were carried out at different locations of two flat surfaces, produced by electrochemical machining under the same conditions. The objective was to find out if the microhardness profile changes its shape along the surface. The investigation had to be made on flat surfaces for which a possible influence of the curvature of the surface on the microhardness profile can be ruled out.

The results in Fig. 5, Fig. 6 and Fig. 7 demonstrate that the extent of the surface softening by electrochemical machining depends strongly upon the region in which the microhardness measurements are made. At a given distance from the machined surface, the microhardness appears to have the same value only in a narrow region with a width of a few diameters of the indenter (compare Fig. 3). The material zone below the machined surface is not altered in a homogeneous way.

It is likely that the microhardness profile results from the defect structure in the given region. This conclusion is supported by the other studies of the Hastelloy X alloy specimen. Changes of the chemical composition, regarding the main constituents of the alloy, could not be detected by the thermal probe traverse, microprobe scans and the scanning microscope. The scanning microscope studies did not reveal evidence for phase or structural changes. Both the microprobe and the scanning microscope are not sensitive enough to give information on the defect structure.

Another interpretation of the microhardness profiles can be based on a generalized type of Rebinder effect. Oxygen from the atmosphere in which the microhardness measurements were made, may interact differently by chemisorption or oxide formation at various spots of the surface for the hardness measurements. The different interaction products in turn lead to different values for the microhardness. At present it cannot be decided if the defect structure in the region of the measurement or a generalized Rebinder effect are responsible for the microhardness profile.

The described results were obtained on specimens machined in sodium chloride solution. If the defect structure of the alloys is responsible for the local softening below the surface, the influence of the type of anion should be small. On the other hand, a large influence might be expected for a generalized type of Rebinder effect because the high rate metal dissolution in the so-called transpassive region will be strongly affected by the anion.

Although embedded particles, probably carbide particles, are seen in scanning microscopy pictures, their average density is about the same over the cross-section of the mounted specimens. Since microhardness measurements were avoided in the vicinity of these particles, they are not considered responsible for the hardness changes.

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