INVESTIGATIONS ON THE PARAMETERS CONTROLLING ELECTROLYTIC POLISHING OF POLYCRYSTALLINE TITANIUM

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Abstract

Polishing of high purity titanium (99.994%) was carried out, overcoming many a hurdle in the process. After having employed various techniques and varied parameters, mirror like finish in the polished specimens was achieved. This could become possible only when the material was subjected to electrolytical polishing. Rod specimens and the mounted ones were electrolytically polished in a bath consisting of H_2SO_4 90% solution, density 1.84(25 vol %), HF 40% solution, density 1.10 (15 vol %) and glacial CH_3COOH 100% solution, density 1.05 (60 vol %), with an additional agent cetyltrimethyl ammonium bromide (0.1-0.5 gml^{-1}), for subsequent tensile testing and microstructural studies.

Introduction

As the 22nd element "Ti" in the periodic table, it is not a rare natural deposit but rather the 4th most common element accounting for 0.6 % of the earth's crust [1,2] Titanium is a multifunctional material, securing its future in a steadily growing market. Where other materials fail, Titanium is a real solution. Titanium and its alloys exhibit a unique combination of mechanical and physical properties and corrosion resistance which have made them desirable for critical, demanding aerospace, industrial, chemical and energy industry service. Of the primary attributes of these alloys, titanium's elevated strength-to-density ratio represents the traditional primary incentive for selection and design into aerospace engines and airframe structures and components. Its exceptional corrosion/erosion resistance provides the prime motivation for chemical process, marine and industrial use. In a nutshell: Titanium is light, strong, corrosion-resistant and biocompatible. All this gives Titanium, its established uses and ever emerging applications, great prospects far beyond the New Millennium!

The material used in the present research work is high purity titanium (99.994%) provided by Alfa Aesar. To begin with, specimens were prepared for tensile testing and microstructural analysis. Rod tensile specimens (length=60mm, thickness=2mm) were machined from as received pure titanium rod of 100 cm length and 2mm thickness while metallographic specimens measuring 15mm in length were also cut from the same rod. All the specimens were annealed in vacuum at 704^0C (1300F) for two hours to relieve internal stresses [3, 4]. The metallographic specimens, however, were annealed prior to mounting, grinding and electrolytic polishing, to keep mounting material, bakelite from any damage during annealing. The tensile specimens, on the other hand, were electrolytically polished in the annealed state.

Experimental Work

Specimen Preparation

The sequential order of preparation method was:

Cutting
Mounting
Grinding and polishing
Annealing

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Cutting

Specimens 6cm long were cut from as-received rods for tensile testing purpose and 1.5cm long for microstructural studies. Maximum care was exercised, as due to its high ductility titanium can easily overheat during cutting and large burrs can occur [4].

Mounting

Small specimens generally require mounting, so that specimen is supported in a stable medium for grinding and polishing. Specimens of irregular shape, great fragility or very small size are best mounted in plastic. Specimens can be mounted quickly by using some thermosetting substance, such as bakelite or, alternatively, a transparent thermoplastic material. These substances mould at about 150^0c, which is usually too low a temperature to cause any structural change in the specimen. In the present research work Bakelite powder was used as mounting material. Specimens were mounted by Prestopress-3 (Model: 4415115, Struers, Denmark) mounting machine at about 150^0C which is too low a temperature to cause any structural change in the specimens.

Grinding and Polishing

Grinding is the most important operation in sample preparation. It is first necessary to obtain a reasonably flat surface on specimen. This can be done either by using a fairly coarse file or by using a motor-driven emery belt. What method is used, care must be taken to avoid overheating the specimen since this may lead to alterations in the microstructure. Specimen should be thoroughly washed in order to prevent carry-over of fillings and dirt to the polishing paper. Emery or silicon carbide cloths and papers are normally used. However, pure and softer titanium, and mounted specimens should always be ground using silicon carbide paper [5,6] In this work five grades (220, 320, 500, 1000 and 1500, from coarse to fine) of silicon carbide (SiC) papers were used. All grinding was done wet. Wet grinding minimizes loading of the abrasive with metal removed from the specimen being prepared. Water flushes away most of the surface removal products. Another advantage of wet grinding is the cooling effect of the water.

When the specimen appears to be flat over the entire surface and grinding has removed all surface imperfections, this operation is considered to be completed. Polishing is the final step in producing a surface that is flat, scratch free and mirror like in appearance. Such a surface is necessary for subsequent accurate metallographic interpretation both qualitative and quantitative. The precautions for cleanliness must be strictly followed. Contrary to the usual procedure of using finer and finer diamond paste for polishing, as is the case in most of the metals, diamond polishing actually introduces continuously mechanical deformation which leaves scratches and smearing on the surface. Therefore diamond polishing should be avoided, especially with polycrystalline high purity titanium. Its extreme ductility makes titanium prone to mechanical deformation and scratching [7], which necessitates a chemical-mechanical polish or electrolytic polish, but the latter gets precedence over the former due to following reason s: speed of results, ease of operation, reproducibility. Also, electrolytic polishing leaves no mechanical deformation on the surface. With these ends in view, both rod specimens and embedded specimens were electrolytically polished, making use of the arrangement as shown below:

Embedded Specimen

Rectifier Facilities

The Electrolyte consisted of H_2SO_4 90% solution, density 1.84 (25 vol %), HF 40% solution, density 1.10 (15 vol %), and glacial CH_3COOH 100% solution, density 1.05 (60 vol %), with an additional agent cetyltrimethyl ammonium bromide (0.1-0.5gml^{-1}) Different arrangements were used for polishing rod like specimens and the embedded ones. The rod specimens were held by small crocodile clips for dipping in the bath and the mounted specimens were clawed by large crocodile clips. Please be aware that the polishing time can vary depending on the purity of titanium and the area of the specimen's surface [7].

Annealing

Annealing refers to a heat treatment in which a material is exposed to an elevated temperature for an extended time period and then slowly cooled. During heat treatment, titanium can react with hydrogen, oxygen and nitrogen. Hydrogen absorption begins at temperatures of 500^{0}C upwards. At temperatures above 700^{0}C, oxygen and nitrogen lead increasingly to the formation of scale layers, with oxygen also diffusing into the work piece surface (diffusion zone). These chemical reactions lead to reduced toughness and thermal stability of the titanium materials. To avoid such air contaminations during annealing, it was necessary that annealing must be carried out in vacuum. Vacuum annealing of Ti reduces hydrogen embrittlement of the metal [8, 9]. With this end in view, specimens were sealed in silica tubes under a vacuum of the order of 10^{-6} torr so that no oxidation or any other reaction can take place during annealing. The arrangement of vacuum system, shown in figure ( ), had following components:

I. Rotary pump
II. Diffusion pump
III. Liquid Nitrogen trap
IV. Silica tubes
V. Tubes sealing arrangements

For heat treatment, a micro controller based muffle furnace, Naberthertm (Model LHT 02/ 18 Germany) was used.

Vacuum sealed specimens were placed in the furnace to be annealed at 704^{0}C for two hours.

Results and observations

The optimal results were attained when the parameters highlighted in the table below were adhered to:

Table 1

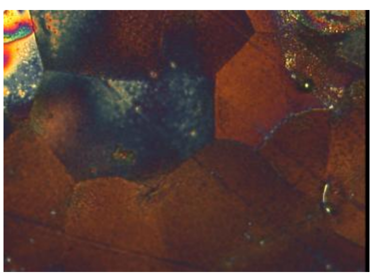
Step	PG 1	PG 2	PG 3	FG 1	FG 2
surface	Sicpaper#220	Sic-paper#320	Sicpaper#500	Sicpaper#1000	Sic-paper#1500
Lubricant	Water	Water	Water	Water	Water
Rpm	300	300	300	300	300
Force[N]	By hand	By hand	By hand	By hand	By hand
Time	130 sec.	100 sec.	80 sec.	60 sec.	60 sec.

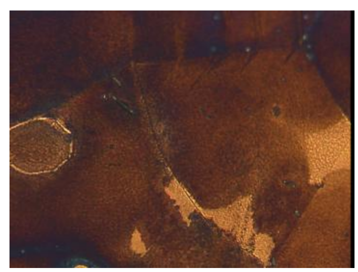
Electrolytic Polishing

Equipment:	Rectifier(100V)
Electrolyte:	As described
Temperature:	15-18^{o}C
Current:	.015A
Voltage:	11V
Time:	15-18 sec

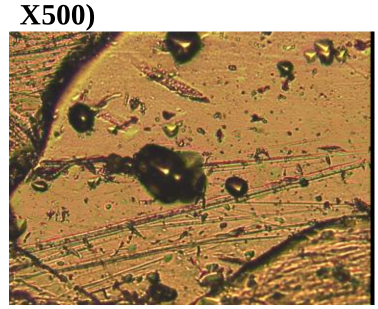
Embedded specimens yielded the best possible elctropolishing results when large crocodile clips were used, as only in that case the desired contact could be established with the mounted part to be polished. The optimal values of applied voltage, bath temperature and time duration came out to be equal to 11V, 16^{0} C, and 15 sec respectively. Rod-specimens were polished making slight adjustments in the parameters employed in case of the mounted ones. Here voltage remained the same, while bath temperature and polishing time were 18^{0} C and 18 sec respectively. Small crocodile clips were used for effectively holding the immersed specimens during electrolytical polishing.

Cetyltrimethyl ammonium bromide was used as an additional agent. This agent modifies, the polarization of one of the two electrodes (alternate adsorption and desorption phenomena) in the medium and leads to modifications in the double-layer phenomena. As a result, the quality of the polishing is improved and less material is removed S. The agent not only provided requisite impetus to the polishing process but also brought added lustre to the polished material. With this bath composition the polished material conspicuously revealed the grains even without etching.





Grains of Titanium (Magnified X500)



Embedded Irradiated Specimen

Deformed Irradiated Specimen

Conclusion

All customary polishing techniques like diamond polishing, chemical mechanical polishing did not produce desired results in case of high purity Ti. As an alternative to chemical-mechanical polishing, elecrolytical polishing was resorted to. The optimum parameters were devised, at Pakistan Council of Scientific and Industrial Research (PCSIR) to obtain a mirror finished surface.Maintining the bath temperature with in the prescribed values posed another problem. This was, however, overcome by employing a two-pronged strategy: Preparing three to four electrolytes at one time, using one of them while keeping the remaining ones in ice tub. Two sets of crocodile clips were used for electrolytic polishing of rod tensile specimens and embedded ones: small size clips for holding the former and large size for holding the latter in the bath. Similarly time duration rationalized for polishing two kinds of specimens, a result of repeated and concerted efforts, turned out to be 15 and 18 seconds in respect of mounted and rod tensile specimens respectively.

As hard-work seldom goes waste; in this case too the strenuous efforts did pay off when the polished specimens revealed the grains quite noticeably.

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