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Low temperature carburised austenitic stainless steel for metal-on-metal tribological contact

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ABSTRACT

S-phase layers formed on biomedical grade austenitic stainless steels have demonstrated significantly enhanced in-vitro wear and corrosion behaviour. To date, most of these tribo-corrosion studies on S-phase treated alloys were conducted using a polycrystalline alumina or cemented tungsten carbide ball as the counterface material. Testing S-phase against S-phase is both scientifically interesting and technologically important in view of their potential applications for the articulating surfaces of metal-on-metal joint prostheses. In this work, biomedical grade 316LVM discs together with AISI 316 balls were low temperature carburised. In-vitro corrosion-wear testing using an S-phase engineered ball against an S-phase engineered disc was performed. Such testing was also complemented with electrochemical impedance spectroscopy, potentiodynamic and cytotoxicity tests. The results have shown that the carburised 316LVM alloy was found to have good in-vitro cytocompatibility and an augmented corrosion and corrosion-wear resistance when compared with the untreated alloy.

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1. Introduction

The favourable corrosion resistance [1–3] and increased surface hardness of carbon S-phase imparted on austenitic stainless steels have already been widely reported in other literature [4]. Studies have also shown that it is possible to produce carbon S-phase layers on medical grade austenitic stainless steel and that there is a possibility for these to be used in orthopaedic applications [1,5,6].

Within the biological environment the material must not illicit an adverse host response by leaching toxic ions by corrosion or corrosion-wear. The interaction among abrasion, rubbing, impact and corrosion can significantly increase total material losses in aqueous environments, thus producing a synergistic effect. The term synergy refers to the enhancement of wear due to corrosion and the change in corrosion rate due to wear. Negative synergism (or antagonism) occurs when the corrosion product during wear provides better protection than the initial surface; an example would be the formation of adherent oxide scale during sliding wear. It is therefore very important to study and in-vitro test newly formed layers holistically in terms of cytotoxicity, corrosion and corrosion-wear response.

The four most common hip joint replacement implants are: polymer-on-metal, ceramic-on-ceramic, polymer-on-ceramic and

metal-on-metal [7]. This means that there is an industrial, medical and scientific interest in the testing of corrosion-wear of metal-on-metal systems. It was proved that carbon S-phase can improve the corrosion-wear response of austenitic stainless steel, however it has always been tested against inert counterfaces. Only one work by Buhagiar et al. [6] has investigated corrosion-wear with both the tribopairs made of the carbon S-phase treated alloy and concluded that there is a huge improvement in wear loss when compared to an untreated-on-untreated system. This improvement was due to a change in wear mechanism from adhesive to mild abrasive wear following S-phase engineering. The combined wear of S-phase against S-phase was close to that of the cobalt-based tribopair (material of choice in metal-on-metal hip joint replacement) under reciprocating sliding wear conditions in Ringer's solution. However the work by Buhagiar et al. [6] does not make use of electrochemical techniques in order to calculate the percentage of material loss due to synergy [8].

Independent studies have confirmed that a carbon rich S-phase results in an improvement of the hardness [4] and wear [9] and corrosion-wear [6,10] system response without any detriment to the corrosion resistance [1–3,11–13]. However no work has been done to evaluate the S-phase cytocompatibility except for two studies on low temperature carburising of: austenitic stainless steel [5]; and cobalt-chromium-molybdenum [12]. Both studies [5,12] have confirmed that carbon S-phase layers formed on these two materials did not reduce the biocompatibility of their respective untreated alloys.

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This work presents a holistic view in terms of material characterisation of carbon S-phase formed on austenitic stainless steel in terms of its suitability in the multidisciplinary field of biomedical orthopaedic implants. It characterises the surface of a carburised stainless steel and then analyses its corrosion, corrosion-wear and cytocompatible response in an in-vitro environment.

2. Material and methods

2.1. Test materials

The materials used in this study consisted of AISI 316LVM stainless steel for surgical implants as per ASTM F138 supplied by L. Klein SA (Switzerland) as rolled round bars of diameter 25.4 mm and AISI 316L supplied by Spheric Trafalgar Ltd. (UK) in spherical form having a diameter of 12.7 mm. The chemical composition of the material in either form is known through a material certificate and is represented in Table 1.

The as-supplied AISI 316LVM bar material was turned on a lathe to two different diameters 25.00 mm and 9.52 mm and cut into coupons of 6 mm and 2 mm thickness respectively. Some of the spheres, to be used for characterisation, had two caps on opposite sides ground off. The flat surfaces of the coupon material were wet-ground using silicon carbide paper from P120 to P1200 grit. The ground surfaces were polished using 6 μm and 3 μm diamond pastes (MetPrep UK) in conjunction with their respective polishing cloths. An ultrasonic bath in acetone and successive drying in hot air was employed prior to every polishing stage, as well as at the end to remove any surface contaminants.

The polished 25.00 mm and 9.52 mm diameter disc coupons, the as-received spheres and the capped off spheres were subjected to a low temperature carburising diffusion-based surface treatment by Bodycote Specialist Technologies GmbH (Landsberg, Germany). During this diffusion surface treatment, the samples were exposed to temperatures below 500 °C and high carbon potentials for several days to obtain a precipitate free S-phase layer.

2.2. Positive control materials

As a control material for the metal-on-metal corrosion-wear tests a Cobalt-Chromium-Molybdenum (CoCrMo) alloy was selected. The 25 mm diameter control disc coupons were made from a wrought un-annealed CoCrMo surgical implants alloy (Biodur® CCM) as per ASTM F1537, supplied by L. Klein SA (Switzerland). The 12.7 mm diameter control balls were made from sintered CoCrMo Stellite® 21, supplied by Kennametal (Italy). The chemical composition of the material in either form is known through a material certificate and is represented in Table 2, mirroring respective material certification.

As a positive control for the cytocompatibility experiments Thermanox™ Coverslips (Thermo Scientific™ Nunc™, USA) were used. These are polyester film strips with very low oxygen content having a hydrophilic surface to increase cell adherence. These films were laser cut into discs 9.52 mm in diameter.

2.3. Preliminary material characterisation

The material characterisation mentioned in this section was performed on low temperature carburised 25 mm diameter austenitic

Table 2

Compositions of the CoCrMo (control) materials used, wt%.

| | | C | Si | Mn | Fe | W | Mo | Ni | Cr | Co |
|--------|--------------|------|------|------|------|------|------|------|-------|------|
| Disc | ASTM F1537 | 0.05 | 0.62 | 0.80 | 0.20 | – | 5.46 | 0.07 | 27.64 | Bal. |
| Sphere | Stellite® 21 | 0.23 | 1.50 | 0.45 | 0.80 | 0.13 | 5.50 | 2.80 | 26.80 | Bal. |

stainless steel coupons and capped off austenitic stainless steel spheres which were cleaned with acetone in an ultrasonic bath.

Standard procedures were followed to prepare metallographic specimens to be examined under a Nikon OPTIPHOT-100 optical microscope. In order to image the S-phase layer created in the carburised 25 mm diameter disc coupon, sectioning normal to the flat surface was performed. This was followed by mounting in Struers Polyfast mounting resin. On the other hand the capped off carburised sphere coupon was mounted on a Struers Taper section with an angle of 10° and then cold mounted in resin. The two mounted specimens were then ground to a P1200 grit using silicon carbide abrasive paper. Samples were then initially polished using 6 μm and 3 μm polycrystalline diamond paste (MetPrep UK) followed by a final polish using chemically active suspended aluminium oxide abrasive (Struers OP-AA). The surfaces were then chemically etched in a solution containing 50 ml of HCl (39% conc.), 25 ml of HNO₃ (69% conc.) and 25 ml of distilled water.

Chemical characterisation of carburised 25 mm austenitic stainless steel disc coupon specimens was accomplished via a LECO GDS-750 QDP Glow Discharge Optical Emission Spectroscopy (GDOES) machine. Calibration was first conducted such that the equipment was set up for all the alloying elements present in the stainless steel with particular attention to carbon. This type of analysis consented for the measurement of the carbon content at increasing depth from the surface of the carburised coupons.

The phase constituents in the untreated and carburised austenitic stainless steel disc and capped off sphere coupons were studied by means of Glancing-Angle X-Ray Diffraction (GAXRD) using a Rigaku Ultima IV diffractometer (CuK α radiation, $\lambda = 0.154 \text{ nm}$) operated in Grazing Incidence Asymmetric Bragg (GIAB) geometry. The incident angle used was set at 3° with a sampling width of 0.05°, and a scan speed of 0.8 s/step in the 30° to 100° 2 θ range. The tube acceleration voltage applied was 40 kV while the current was 30 mA.

Microhardness indentations were carried out using a Mitutoyo MVK-H2 with a Vickers hardness indenter. Ten repeated indentations were made on the surface at a load of 100 gf and care was taken to ensure that each indent was at least 5 diameters away from the previous indent.

In order to obtain a hardness versus depth profile of the carbon diffusion layer formed, the austenitic stainless steel disc and capped off sphere were cold mounted on taper section angles (Struers). This inclined the sample by 10° and therefore provided more space for indentations since the layer's depth was magnified by an order of 5. The mounted samples were metallographically polished and nano-indentation was carried out on the magnified treated layer using a Micromaterials NanoTest (UK) equipped with a diamond Berkovich indenter at a load of 100 mN. Each indent was carried out by using computer control to start at a load of 0.1 mN and gradually increasing by a rate of 2 mN s⁻¹ until the pre-set load was reached. The load then ramped down at the same rate of 2 mN s⁻¹. A set of 25 indents was performed, with the first indent effected at the surface of the sample layer. The location of indents was set such that every subsequent indent

Table 1

Compositions of the stainless steel materials used, wt%.

| | | C | Si | Mn | P | S | N | Cu | Mo | Ni | Cr | Fe |
|--------|--------|-------|------|------|--------|-------|-------|------|------|-------|-------|------|
| Disc | 316LVM | 0.011 | 0.25 | 1.69 | 0.016 | 0.003 | 0.088 | 0.03 | 2.78 | 14.56 | 17.33 | Bal. |
| Sphere | 316L | 0.08* | 1.0* | 2.0* | 0.045* | 0.03* | – | – | 2–3 | 10–14 | 16–18 | Bal. |

* Represents maximum value.

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