



4th Symposium on Surface Hardening of Corrosion Resistant Materials VALLETTA, MALTA 22nd – 24th June 2016

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4th Symposium on Surface Hardening of Corrosion Resistant Materials

University of Malta Valletta Campus, St Paul Street, Valletta, Malta 22-24 June 2016

Low-temperature (below 500°C) interstitial treatment of corrosion-resistant alloys is a developing technology in surface engineering, resulting in performance enhancements in hardness and wear, corrosion-wear, fatigue and corrosion resistance. When applied to austenitic stainless steels, the resultant surface treatment has been described as S-phase, or expanded austenite. Major advances in understanding the technology and its application to austenitic, ferritic, martensitic and duplex alloys have occurred since the last symposium on this topic, held in Pittsburg in October 2014. This program will serve as a "state of the art" meeting, bringing together researchers from universities and government agencies all over the world, as well as providers and end users of low temperature surface hardening techniques. Papers devoted to all aspects of research on low-temperature processes (gas, ion and plasma processes) for surface enhancement of corrosion resistant alloys (Fe-, Ni-, and Co-based Cr-containing alloys, as well as Ti and other passive alloys) by interstitial hardening (carburizing, nitriding and nitrocarburizing) will be presented. Specific sessions include:

- Fundamentals and processing of S-phase
- Properties and performance of S-phase
- Use of S-phase for biomedical applications
- Stability and degradation of S-phase
- S-phase on Duplex stainless steel and Ni-based super alloys
- Diffusion treatments Ti-, Zr- and Fe-based alloys

Scientific Committee

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Kathy Murray

ASM International

Kelly Thomas

ASM International

Programme

Wednesday 22nd June 2016

08:00 - 09:00

Opening of the symposium		
Marcel Somers (Invited)	Expanded austenite; from fundamental parameters to predicting concentration- and stress-depth profiles	
Santiago Corujeira Gallo	Surface hardening of austenitic stainless steel in segmented hollow cathode plasma	
Coffee break		
Andreas Karl	Low Temperature Surface Hardening of Maraging steels	
Ulli Oberste-Lehn	Laser processing of low temperature surface hardened stainless Steel	
Lynne Hopkins	Structure and mechanical properties of nitrogen- containing Austenitic manganese coatings deposited by sputter PVD	
Lunch break		
performance of S-phase (Sessi Hisao Fujikawa (Invited)	on chair: Dr Ing. Joseph Buhagiar) Corrosion mechanism of Austenitic stainless steels with Sphase produced by Carburising and Nitriding at low temperature, respectively Property enhancement of 303 austenitic stainless steel by	
Aladyllig Li	low-temperature plasma nitriding	
Coffee break		
Yong Sun	Response of nitrogen S-phase layer and carbon S-phase layer on stainless Steel to 1D and 2D sliding wear	
Xiao Tao	Triode plasma nitriding of Hadfield Austenitic manganese	
	Marcel Somers (Invited) Santiago Corujeira Gallo Coffee break Andreas Karl Ulli Oberste-Lehn Lynne Hopkins Lunch break sion: performance of S-phase (Sessi Hisao Fujikawa (Invited) Xiaoying Li Coffee break	

Thursday 23rd June 2016

08:00 - 09:00

Use of S-phase			
09:00 - 09:55	Joseph Buhagiar (Invited)	Low temperature carburising of AISI 316: A biological, tribological and electrochemical interdisciplinary approach	
09:55 - 10:20	Bertram Mallia	Tribocorrosion response of PVD CrN-coated low temperature carburised implant grade 316LVM	
10:20 - 10:50	Coffee break		
10:50 - 11:15	Yangchun Dong	Friction property of low temperature plasma carburised micro-patterning of ASTM F1537 CoCrMo alloy	
11:15 - 11:40	Joseph Buhagiar	Low temperature carburised CoCrMo: A biological and electrochemical interdisciplinary approach	
12:00 - 13:30	Lunch break		
Afternoon sess Stability and De	ion: gradation of S-phase (Session	Chair: Prof Marcel Somers)	
Stability and De	gradation of S-phase (Session		
		Stability and environmental degradation of S-phase Thermal stability of Expanded Austenite in a 316L	
Stability and De 13:30 - 14:25 14:25 - 14:50	Hanshan Dong (Invited) André Paulo Tschiptschin	Stability and environmental degradation of S-phase Thermal stability of Expanded Austenite in a 316L Austenitic stainless Steel	
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Stability and De 13:30 - 14:25 14:25 - 14:50 14:50 - 15:15 15:15 - 15:40	Hanshan Dong (Invited) André Paulo Tschiptschin Xiaoying Li Luis Armando Espitia	Stability and environmental degradation of S-phase Thermal stability of Expanded Austenite in a 316L Austenitic stainless Steel Response of SN/C-Phase to rapid Annealing Linear scratch tests of active screen low temperature	
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Friday 24th June 2016

08:00 - 09:00	Pogistration	
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Morning session Diffusion treatm		loys (Session chair: Prof Marcel Somers)
09:00 - 09:25	Nils Elmegaard-Fessel	Surface hardening of duplex stainless steel by high- temperature solution nitriding with/without subsequent low temperature surface hardening
09:25 - 09:50	Zhenxue Zhang	Surface co-alloying of corrosion resistant CM247LC superalloy with V/Ag and N
09:50 – 10:15	Thomas Strabo Hummelshøj	Application of next-generation stainless steel & titanium surface hardening
10:15 – 10:45	Coffee break	
10:45 – 11:10	Glenn Cassar	PIRAC nitriding of titanium alloys
11:10 – 11:35	Thomas Christiansen	Gaseous surface hardening of titanium and titanium alloys
11:35 – 12:00	Abdulkarim Alansari	Effect of oxidation temperature and surface roughness on tribological behaviour of thermally oxidized pure zirconium
12:00 - 12:25	Yan Jing	Phase transformations during low temperature nitrided Inconel 718 superalloy
12:25 – 13:00	Closing session chaired by Prof Marcel Somers	
13:00 - 14:30	Lunch break	

Social Programme

Wednesday 22 June 2016 at 18:30

Optional Three Cities Evening Tour & Dinner

The tour will start with a crossing from Valletta to the Three Cities by boat, a splendid way of experiencing the Grand Harbour. Upon arrival in Vittoriosa (or Birgu, as it is better known), the tour proceeds with a walk along the streets of this city. The name 'Vittoriosa' derives from the vital role the city played in the Great Siege of 1565, namely the victory of the Knights of St John and the Maltese against the Ottoman forces. A monument commemorating this victory can be admired in the main square of the city.

Vittoriosa was the area first selected by the Knights to serve as headquarters of the Order, before the building of the city of Valletta. In fact, the first buildings erected by the Knights are to be found here. Practically none of these building, however, are intact as the area was heavily damaged during the Second World War. Some of the old buildings have been totally rebuilt whilst others have been restored and are nowadays residences, offices or restaurants. The evening tour ends in one of these restaurants (Tal-Petut) for an exquisite dinner.

Thursday 23 June 2016 at 17:45

Mdina Evening Walking Tour & Conference Dinner

Participants will be taken from the Conference venue at the Valletta Campus to visit the Department of Metallurgy and Materials Engineering Labs at the University of Malta, Msida Campus. They will then proceed to Mdina and Rabat for the conference dinner.

Mdina, the old capital city of Malta, is the Island's most picturesque city. The tour starts with a walk through the narrow streets of this small medieval city, which allows the visitors to take in the highly ornate façades of the palazzi belonging to the most important Maltese noble families. The oldest palazzo, Palazzo Santa Sofia, dates back to the 15th century. Dominating the city is the imposing Cathedral built by the famous Maltese architect Lorenzo Gafá. The dome of this sacred building was Gafa's masterpiece and is now considered as one of the symbols of the city.

The walk leads to the bastions, one of the highest points in the area, from where visitors can admire a panoramic view of the Island. Overlooking the whole area, it is easy to understand why the medieval soldiers used this part of the city as a look-out post in order to intervene should there be attacks in the surrounding areas. The walk is followed by dinner in Da Luigi Restaurant in Rabat.

ABSTRACTS

Wednesday 22 nd June 2016		
08:00 - 09:00	Registration	
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09:00 - 09:25	Opening of the symposium	
Morning session Fundamentals and processing of S-phase		
Session chair:	Prof Hanshan Dong	

Session chair: Prof Hanshan Dong

09:25 - 10.20 Invited #1

Expanded austenite; from fundamental parameters to predicting compositionand stress-depth profiles

Marcel A.J. Somers 1*

¹Technical University of Denmark, Produktionstorvet b.425, DK 2800 Kongens Lyngby, Denmark *somers@mek.dtu.dk

The case developing during low temperature surface hardening of austenitic stainless steel by nitriding, carburizing or nitrocarburizing consists of a supersaturated interstitial solution of nitrogen and/or carbon in austenite. The favourable properties of this so-called expanded austenite depend on the profiles of interstitial concentration and associated composition-induced residual stress over the case. The prediction of composition and stress profiles for a certain steel grade from process parameters as gas composition, temperature and time would enable targeted surface engineering of stainless steels. Furthermore, such a numerical model would enable the design of stainless steel compositions that are tailored for optimal performance during low temperature surface hardening.

In the present contribution a numerical model is presented to predict the interdependent composition and stress profiles over the expanded austenite case as developing during low temperature nitriding of austenitic stainless steel. The model departs from fundamental parameters as determined experimentally on homogeneous powders and foils and a diffusion model that incorporates the interaction of composition and stress as well as the elastic-plastic accommodation of composition-induced lattice expansion in the case.

The presentation showcases research results of the last 16 years and is the joint achievement of many co-workers. The presentation includes the following topics:

- Interstitial solubility, interstitial diffusion and phase stability of homogeneous expanded austenite;
- Crystallography, thermal expansion and magnetism of homogeneous expanded austenite;
- Residual-stress determination in expanded austenite cases;
- Numerical modelling aspects of composition- and stress profiles.

10:20 - 10.45 Oral #1

Surface hardening of austenitic stainless steel in segmented hollow cathode plasma

Santiago Corujeira Gallo 1*

¹ School of Metallurgy and Materials, The University of Birmingham, Birmingham B15 2TT,UK ^{*} corujeis@bham.ac.uk

The hollow cathode effect has received much attention over the years, because of its detrimental implications for plasma treatments as well as its potential uses in surface engineering. This type of electric discharge takes place when the sheath of two cathodic surfaces overlap, thus creating an intense plasma discharge and high current density over a relatively small area. In contrast, the segmented hollow cathode (SHC) geometry consists of alternated anodes and cathodes. In such arrangement, the intensity of the plasma is modulated by the geometrical arrangement of electrodes as well as the plasma conditions, namely the gas mixture, the gas pressure and the applied potential. This study explored the use of SHC for surface hardening of austenitic stainless steel AISI 316. The samples were plasma nitrided in SHC arrangements at low temperature, to form expanded austenite. In addition, electrodes of different materials were used in the SHC setup, for surface alloying or coating. The microstructure and the layer thickness of the SHC plasma treated specimens were assessed by optical microscopy (OM) and scanning electron microscopy (SEM), the present phases were identified by X-Ray diffraction (XRD) and the surface hardness was assessed by micro- and nano-indentation experiments. The results are compared with the more established DC and Active Screen plasma techniques. The opportunities and limitations of the SHC arrangement are critically discussed.

10:45 - 11:15

Coffee break

11:15 - 11:40 Oral #2

Low temperature surface hardening of Maraging steels

Andreas Karl 1* and Ulli Oberste-Lehn 1

¹Bodycote Specialist Technologies GmbH; Landsberg, Germany

*Andreas.Karl@bodycote.com

Maraging steels are a group of low-carbon high-strength steels with good ductility and machinability. The hardening mechanism is based on the formation of a tough and ductile Fe-Ni martensitic matrix which is further hardened by subsequent precipitation of intermetallic phases during age hardening. Mainly Mo and Ti are added as precipitation forming elements. The corrosion resistance of Maraging steels can reach values of standard martensitic stainless steels. Hardness values up to 500-600 HV in aged conditions can be achieved, but may be too low when it comes to adhesive or abrasive wear.

Gas nitriding is a standard process to improve wear and fatigue properties of those materials, but reduces corrosion resistance due to the formation of nitrides. Furthermore, the core hardness of Maraging steels is reduced significantly due to process temperatures above ageing temperatures. With low temperature carburizing and nitrocarburizing (below 500°C) it is possible to improve not only wear properties, but also the corrosion resistance of Maraging steels whilst maintaining the core hardness and strength.

In this paper, the low temperature carburizing and nitrocarburizing of a MARAGE 300 alloy (1.6354) is presented. Technological properties including surface hardness and diffusion depth are presented, as well as corrosion and wear properties. The distribution of interstitial elements is evaluated by means of GDOES measurements. XRD-Patterns show a shift to lower angles without the formation of additional phases, which indicates expansion of the lattice and thus an expanded martensitic structure.

11:40 - 12:05 Oral #3

Laser processing of low temperature surface hardened stainless steel

Ulli Oberste-Lehn 1* and Andreas Karl 1

¹Bodycote Specialist Technologies GmbH; Landsberg, Germany

*Ulli.Oberste-Lehn@bodycote.com

Low temperature surface hardening of stainless steel has evolved in industrial applications for over 25 years. With increasing demand, especially in the automotive industry, as well as for medical applications or applications in hazardous environments, the traceability of products becomes ever more vital. A solution for providing traceability is individual marking of every part or batch by means of laser marking.

In this paper three different types of laser marking are presented. First, feasible laser parameters are assessed. The optical appearance of the marking and the corrosion resistance are compared for marking before and after low temperature carburizing. The evaluation will support decision-making on which marking technique should be applied and whether it should be before or after low temperature surface hardening.

Furthermore, especially in the automotive industry, there is a high demand for joining after low temperature surface hardening. Suitable laser welding parameters were evaluated for 316L sheet metal. Those parameters were applied for both low temperature carburized and nitrocarburized 316L sheet metal, showing superior weld seam quality and corrosion resistance for the low temperature carburized specimens. In particular, the high porosity of the weldment limits the use of low temperature nitriding or nitrocarburizing.

12:05 - 12:30 Oral #4

Structure and mechanical properties of nitrogen-containing Austenitic manganese coatings deposited by sputter PVD

Lynne Hopkins 1*; X. Liu 1; C.Liu 1; X Tao 1; Allan Matthews 2 and Adrian Leyland 1

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International Centre for Advanced Materials, School of Materials, University of Manchester, UK

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The use of Nitrogen as an alloying element in steel is either limited to small quantities (<2 wt% / 7.5at%) in bulk materials (where it is used primarily as an austenite stabiliser and mechanical/tribological property enhancer), or introduced in much larger quantities (>14 wt% / 40 at%) in thermochemical surface engineering treatments – where it is used to create a hard, corrosion-resistant diffusion layer of (typically) 20-30 μ m depth (commonly referred to as "Expanded Austenite" or "S-phase").

This study examines the effects of Nitrogen incorporation in a high-manganese austenitic stainless steel (Staballoy AG 17), at levels that lie between the two abovementioned extremities, with the intention of improving the mechanical and wear properties without compromising the inherent high resistance to corrosion and galling wear which such alloys possess.

Thick, dense and featureless coatings of Austenitic-Mn steel containing different levels of interstitial Nitrogen were deposited by reactive magnetron sputtering in an Argon-Nitrogen plasma. The resulting microstructures were characterized by optical micrography, SEM and XRD; the mechanical properties, as analyzed by nanoindentation, indicate that hardness increases with increasing nitrogen content.

12:30 - 14:00

Lunch break

Afternoon session | Properties and performance of S-phase

Session chair: Dr Ing. Joseph Buhagiar

14:00 - 14:55 Invited #2

Corrosion mechanism of Austenitic stainless steels with S-Phase produced by carburising and nitriding at low temperature, respectively

Hisao Fujikawa 1*

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It is well known that S-phase produced on the surface of austenitic stainless steel by nitriding and carburizing at low temperatures, respectively, has good corrosion resistance in acid solution, chloride solution and so on. As one of reasons in which austenitic stainless steels with S-phase show good corrosion resistance, Cr carbides or nitrides do not precipitate into the S-phase. However, the improvement of corrosion resistance is not explained only by this reason. We can produce austenitic stainless steels with S-phase by our unique process in gas nitriding and carburizing, respectively. Using these processes, we have studied corrosion mechanism of austenitic stainless steels with S-phase produced by low temperature nitriding or carburizing, according to electrochemical studies and analysises of passive film and so on. As the results, in the case of the low temperature nitriding, it was found that when the passive film is broken by Cl⁻ ion and so on and then pitting or crevice corrosion occurs, NH⁴⁺ ions are eluted and the corrosion resistance is improved by the control of corrosion environment. In the case of the low temperature carburizing, it was also found that Cr content in the passive film is enriched higher than that in the usual stainless steels and the corrosion resistance of steels is improved by the passive film with higher Cr content. I will show our process and the results of corrosion studies more exactly.

14:55 - 15:20 Oral #5

Property enhancement of 303 austenitic stainless steel by low-temperature plasma nitriding

Xiaoying Li 1*; Marjorie Benegra 2; Yakun Gao 1 and Hanshan Dong 1

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- ² UTFPR Universidade Tecnológica Federal do Paraná, Brazil
- *X.LI.1@bham.ac.uk

303 austenitic stainless steel is designed for mechanical and biomedical components which need to be highly machined, due to the sulphur added intentionally for this purpose. However, the intrinsic low-hardness has limited its wider applications. A possible surface engineering technique to increase the hardness and corrosion resistance is the plasma nitriding. This study intends to improve the corrosion resistance of 303 stainless steel. For that DC plasma nitriding was used at different temperatures (400, 420 and 480 °C) and treatment times (10, 15 and 20 min). After that, the characterization was conducted using scanning electron microscope, glow discharge optical emission spectrometry, X-ray diffraction, Vickers hardness and anodic polarization tests using a 0.1 M NaCl solution. The experimental results have shown that plasma nitriding produced a dense and hard surface layer ranging from 5 to 40 μ m thick. A single S-phase layer was formed during plasma nitriding at 400 °C, which is supersaturated with nitrogen without precipitation. The formation of S-phase layer significantly improves the surface hardness and also effectively addresses the pitting corrosion of free-machining stainless steel.

15:20 - 15:50

Coffee break

15:50 - 16:15 Oral #6

Response of nitrogen S-phase layer and carbon S-phase layer on stainless steel to 1D and 2D sliding wear

Yong Sun 1*

¹ School of Engineering and Sustainable Development, De Montfort University, Leicester, UK *ysun01@dmu.ac.uk

In this work, 316L austenitic stainless steel was plasma nitrided and carburized at low temperatures to produce a nitrogen S-phase layer and a carbon S-phase layer, respectively, on the surface. The tribological and tribocorrosion behaviour of the untreated, nitrided and carburized specimens were investigated under both dry sliding condition and tribocorrosion condition in 0.5M H₂SO₄ solution, using both one-dimensional (1D) and two-dimensional (2D) reciprocating motions. The 2D motion was produced by linearly reciprocating the specimen holder as the primary motion and at the same time reciprocating the ball (pin) holder at a high frequency and small amplitude as the secondary motion. The results showed that 2D sliding had significant effects on the wear behaviour of the test specimens. Under dry condition, 2D sliding let to an increase in the width and depth of the wear track and a significant increase in wear volume by several factors. The nitrogen S-phase layer was very resistant to both 1D and 2D dry sliding wear. While the carbon S-phase layer showed good resistance to 1D dry sliding wear, it experienced accelerated wear during 2D sliding. Under tribocorrosion conditions in 0.5M H₂SO₄ solution, 2D sliding had a significant effect on the evolution of open circuit potential. The carbon S-phase layer was effective in reducing material removal under both 1D and 2D tribocorrosion conditions. On the other hand, the nitrogen S-phase layer exhibited much deteriorated wear resistance during tribocorrosion in the solution. The mechanisms of material removal are discussed in terms of the structures of the S-phase layers and their mechanical and electrochemical responses to the corrosive solution.

16:15 - 16:40 Oral #7

Triode plasma nitriding of Hadfield Austenitic manganese steel

Xiao Tao 1*; John Kavanagh 2; Allan Matthews 3 and Adrian Leyland 1

- ¹ Department of Materials Science & Engineering, The University of Sheffield, UK
- ² Advanced Composite Materials Facility, Dept. of Chemistry, University of Southampton, UK
- ³ International Centre for Advanced Materials, School of Materials, University of Manchester, UK
- *xtao1@sheffield.ac.uk

It has been shown in literature that the addition of nickel retards the nitrogen diffusion rate during low temperature nitriding of chromium-containing austenitic stainless steels. Furthermore, Cr is believed to be an essential element in forming expanded austenite, since it appears to assists in 'trapping' N atoms interstitially in the austenite lattice without forming nitrides (even at ≥ 20 at.% N). However, due to the strong chemical affinity between Cr and N, Cr nitrides might still occur 1) as precipitates in the diffusion zone (which will degrade material corrosion resistance), or 2) as a dense superficial (compound) layer at the surface, which will inhibit further nitrogen inward diffusion. In this case, austenitic materials which do not contain strong nitride-former alloying elements (such as Cr) have the potential to beneficially suppress nitride formation during plasma nitriding - enabling a more efficient nitrogen diffusion treatment, where a deep and highly N-supersaturated austenitic layer can be formed after a relatively short treatment time.

Hadfield steel has a nominal composition of Fe-11Mn-1.1C-0.4Si (without Cr or Ni) and generally maintains its austenitic structure upon sufficiently rapid quenching from (solution treatment) temperatures. Furthermore, N acts in itself as a powerful austenite stabiliser even at low concentrations (~ 3-5 at.%). The nitriding response of austenitic manganese steel is therefore interesting to investigate and, in this study, a standard 11wt.% Mn Hadfield steel is triode plasma nitrided at 300°C for 4, 8, 12 and 20hrs, 500°C for 4 hrs and 700°C for 1 hr to investigate treatment depth, microstructural stability and diffusion hardening effects.

18:30

Three cities optional tour and dinner (Tal-Petut, Vittoriosa) (Meeting at symposium location)

Thursday 23rd June 2016 08:00 - 09:00 Registration Morning Session | Use of S-phase for biomedical applications Session chair: Prof Hanshan Dong 09:00 - 09.55 Low temperature carburising of AISI 316 LVM: A biological, tribological and Invited #3 electrochemical interdisciplinary approach Joseph Buhagiar 1* Department of Metallurgy and Materials Engineering, University of Malta, Msida, MSD2080, Malta *joseph.p.buhagiar@um.edu.mt This study focuses on the bioengineering properties of surface hardened AISI 316LVM (ASTM F138) by low temperature carburising (S³P - Bodycote Specialist Technologies GmbH). The work was prompted by the need to find an improved biomedical alternative for current metal-on-metal articulation prosthesis. The latter are reported to have high failure rates and associated genotoxic and biohazardous effects causing complications that lead to low quality of life for the patients. Studies have been carried out on four main fronts including: material characterisation, "In vitro" corrosion testing, tribocorrosion testing and solid state biological testing. The corrosion response was investigated via potentiodynamic testing and Electrochemical Impedance Spectroscopy (EIS) in deaerated Ringer's solution. The results have shown that the carburised 316LVM alloy was found to have an augmented corrosion resistance when compared with the untreated alloy. This was attributed mainly to the more resistive passive film developing on the surface of the carburised alloy. The low temperature carburised stainless steel was also found to perform as well as its untreated cytocompatible counterpart when regarding direct "in vitro" contact cytotoxicity carried out using a human foetal osteoblast (hFOB 1.19) cell line. It was noted that the combined effect of higher wetability and an increase in surface roughness of the carburised alloy produced no effect on the biocompatibility of the alloy, or rather each parameter having an effect which is countered by the effects of another parameter. To date, most of these tribocorrosion studies on S-phase treated alloys were conducted using a polycrystalline alumina or cemented tungsten carbide sphere 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-onmetal joint prostheses. In this work, biomedical grade 316LVM hemispheres and discs were low temperature carburised. In-vitro reciprocating metal-on-metal corrosion-wear was performed in Ringer's solution and dilute bovine serum at three different potentials: cathodic (-700 mV vs SCE); open circuit and anodic (+100 mV vs. SCE). The carburised-oncarburised tribopair showed an improvement in the tribocorrosion response when compared to untreated-on-untreated stainless steel counterpart. This augmentation was registered in both testing solutions and also at all the three potentials.

09:55 - 10:20 Oral #8

Tribocorrosion response of PVD CrN-coated low temperature carburised implant grade 316LVM

Imer Cardona 1; Joseph Buhagiar 1; Peter A. Dearnley 2; Andreas Karl 3; and Bertram Mallia 1*;

- ¹ Department of Metallurgy and Materials Engineering, University of Malta, Msida, MSD2080, Malta
- ² Boride Services Ltd, Leeds, UK
- ³ Bodycote Specialist Technologies GmbH; Landsberg, Germany
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Austenitic stainless steel, a popular implant material, displays poor tribocorrosion behaviour in sliding contacts. When sliding against smooth conformal surfaces in corrosive environments this material displays increased surface roughening and significant material loss by passive film damage and regeneration mechanism (Type I corrosion-wear).

This paper investigates how the tribocorrosion of medical grade 316LVM could be improved by the application of duplex CrN hard coatings on low temperature carburised surfaces. The role of the low temperature carburising on the performance of the duplex treatment under simultaneous mechanical and corrosion actions was investigated. The low temperature carburising treatment resulted in a thick expanded austenite (S-Phase) layer which enhanced the damage tolerance resistance of the magnetron sputtered CrN PVD coating when subjected to nano-scratch testing. Following excellent response to static corrosion testing, the treated surfaces were investigated for their tribocorrosion performance. Reciprocating sliding tribocorrosion tests were carried out against a smooth alumina ball in Ringer's solution under different electrochemical conditions. The duplex layer displayed high resistance to type I damage. The low temperature carburised layer prevented substrate localised corrosion attack during tribocorrosion testing. More work needs to be done to better understand the mechanism behind CrN PVD coating delamination during tribocorrosion testing despite their good adhesion to the underlying S-phase layer.

10:20 - 10:50

Coffee break

10:50 - 11:15 Oral #9

Friction property of low temperature plasma carburised micro-patterning of ASTM F1537 CoCrMo alloy

Yangchun Dong 1*; Petr Svoboda 2; Ivan Krupka 2 and Hanshan Dong 1

- ¹ School of Metallurgy and Materials, The University of Birmingham, Birmingham B15 2TT,UK
- ² Brno University of Technology, Brno, Czech Republic
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Corrosion-wear and iron-release are two common problems that affect the biocompatibility of the materials of hip joint prosthesis. The solution to this main challenge remains to control the following two aspects—the amount of materials shedding from the wearing of counterparts, and the friction/lubrication between the moving components, i.e. femoral head and taper liner. In this study, a plasma case hardened micro-geometrical patterning was created on the CoCrMo surfaces with the possibility of application in long-term MoP prosthesis bearing surfaces. This surface engineered CoCrMo alloy showed improved friction performance compared to the untreated surface wearing against UHMWPE, especially in the start-and—stop of the mixed lubricated regime. The low-temperature plasma carburising generated a uniform S-phase case on the micro-pattered CoCrMo surface. There was no noticeable effect of the internal stress of patterning on the forming of S-phase. As a result, the wear rate of the fragile patterning was increased by three to five orders of magnitude by the sequential treatment of case hardening.

11:15 - 11:40 Oral #10

Low temperature carburised CoCrMo: A biological and electrochemical interdisciplinary approach

Malcolm Caligari Conti ¹; Josianne Cassar ¹; Emmanuel Sinagra ²; Andreas Karl ³; Pierre Schembri Wismayer MD ⁴; Bertram Mallia ¹ and <u>Joseph Buhagiar</u> ¹[†];

- Department of Metallurgy and Materials Engineering, University of Malta, Msida, MSD2080, Malta
- ² Department of Chemistry, University of Malta, Msida, MSD2080, Malta
- ³ Bodycote Specialist Technologies GmbH; Landsberg, Germany
- ⁴ Department of Anatomy, University of Malta, Msida, MSD2080, Malta
- *joseph.p.buhagiar@um.edu.mt

This study focuses on the bioengineering properties of surface hardened Co-Cr-Mo alloys by low temperature carburising. The work was prompted by the need to find an improved biomedical alternative for current metal-on-metal articulation prosthesis. The latter are reported to have high failure rates and associated genotoxic and biohazardous effects causing complications that lead to low quality of life for the patients.

Studies have been carried out on three main fronts including: material characterisation; "In vitro" corrosion testing; and solid state and elution biological testing.

The carburised alloy was seen to gain an increase in hardness of approximately 250% over the untreated alloy and had a layer thickness of $16 \mu m$.

Potentiodynamic tests were performed in phosphate buffered saline solution, Ringer's solution and dilute bovine serum solution mimicking the conditions in vivo as closely as possible. In all solutions, the low temperature carburised Co-Cr-Mo alloy was seen to outperform the untreated alloy. In this regard the low temperature carburised alloy showed a consistently higher OCP value and a lower corrosion current. This was attributed mainly to the more resistive passive film developing on the surface of the carburised alloy. This then gave rise to a better performance in terms of in vitro elution biocompatibility testing performed on the carburised alloy.

The low temperature carburised alloy was also found to perform as well as its untreated biocompatible counterpart when regarding direct "in vitro" contact biocompatibility carried out using a bone osteosarcoma (Saos-2) cell line, a mouse osteoblast (MC3T3-E1) cell line and human foetal osteoblast (hFOB 1.19) cell line as well as primary human osteoblast cells. It was noted that the combined effect of higher wetability and an increase in surface composition of the carburised alloy produced no effect on the biocompatibility of the alloy, or rather each parameter having an effect which is countered by the effects of another parameter.

12:00 - 13:30

Lunch break

Afternoon session | Stability and degradation of S-phase

Session Chair: Prof Marcel Somers

13:30 - 14:25 Invited #4

Stability and environmental degradation of S-phase

Hanshan Dong 1*

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Corrosion resistant alloys have played an important role in many industrial sectors (especially in oil, gas and nuclear applications) to combat environmental degradation. The development of S-phase surface engineering has paved way towards wear and corrosion resistant surfaces for demanding applications. However, S-phase is thermodynamically metastable will change to more stable phases under certain favourable conditions.

This talk overviews the major progress made in advancing scientific understanding of the stability of S-phase and in combating its environmental degradation. It will start with a brief review on the nature and thermal stability of S-phase at elevated temperatures and followed by more recently research into thermos-mechanical stability of S-phase under combined thermal and mechanical conditions. Then, the potential of low-temperature plasma thermochemical treatment in combating environmental assisted cracking of stainless steel is demonstrated by way of example. For instance, within a stress sulphide cracking environment, both the treated precipitation hardened and martensitic stainless steels maintained a 100% pass record, in accordance to British oil and gas sour service standards, where their untreated counterparts failed; following a hydrogen charging experiment low temperature plasma alloying showed a 96% average improvement across all samples in the prevention of hydrogen permeation.

The talk will finish with a discussion on planned international collaboration involving Argentine, Chinese and British researchers in assessing the stability and radiation damage of S-phase for the safe application of S-phase surface engineered stainless steel for the nuclear industry.

14:25 - 14:50 Oral #11

Thermal stability of Expanded Austenite in a 316L Austenitic stainless steel

<u>Hanshan André Paulo Tschiptschin</u>^{1*}; Carlos Eduardo Pinedo ²; Arthur Seiji Nishikawa ¹ and Luis Bernardo Varela ¹

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Expanded austenite formed after low temperature plasma nitriding of austenitic stainless steels is known for its excellent wear and corrosion resistance especially when working in systems where galling, erosion-corrosion, cavitation-erosion and pitting corrosion resistance are a major concern. These wear and corrosion properties may degrade by exposure of the surface hardened steel to high temperatures, between 400°C and 700°C. DC low temperature plasma nitrided 316L austenitic stainless steel (400°C for 20 hours) was heated up to investigate the stability of the 14µm thick expanded austenite layer in the range 400°C < T 700°C. Time-resolved X-ray diffraction experiments were undertaken in a thermomechanical simulator coupled to the Brazilian National Synchrotron Light Source. Two series of experiments were carried out: a) isothermal treatments conducted at 400°C and 550°C, for 30 min and b) a continuous heating experiment from room temperature up to 700°C, with a heating rate of 12°C/min. The results show that during the continuous heating experiment, expanded austenite remained approximately stable up to 500°C, without losing nitrogen and maintaining the expanded austenite lattice parameter. For temperatures higher than 550°C a continuous decrease in expanded austenite lattice parameter occurred and chromium nitride precipitation started by a discontinuous precipitation mechanism; at temperatures greater than 625°C bcc ferrite precipitated accompanied by CrN and Cr2N chromium nitrides and Fe₂₋₃N iron nitride. The initial 0.385 nm expanded austenite lattice parameter increased up to 0.3875 nm at 425°C due to thermal expansion, indicating an average thermal expansion over the 14 µm thick analyzed expanded austenite layer of 5 x 10-5 K⁻¹. This value is much lesser than the values reported for the thermal expansion coefficients of the fcc austenitic phase in austenitic stainless steel K = 15 x 10-5 K⁻¹, indicating that expanded austenite is also losing compressive residual stresses during heating. The isothermal experiments at 400°C showed that expanded austenite is stable, for at least 30 min and expansion of the lattice during heating is compensated by a drift of X-ray peaks due to loss of compressive residual stresses. Isothermal treatment at 550°C showed that expanded austenite loses stability and decomposes to fcc austenite and CrN nitride by a discontinuous precipitation mechanism after 15 min of exposure.

14:50 - 15:15 Oral #12

Response of SN/C-Phase to rapid annealing

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Formation of nitrogen/carbon interstitial supersaturated expanded austenite i.e. S-phase layers on austenitic stainless steels by low temperature nitriding/carburising can significantly improve their hardness, wear resistance and fatigue properties. However, the metastability of the S-phase needs to be considered from both a scientific and technological point of view. Some researches have been carried out to investigate the stability the S-phase by isothermal annealing but little work has been done on the repose of the S-phase to rapid annealing.

In this research, the response of S-phase to rapid annealing has been investigated to advance scientific understating of the metastability of the S-phase and to explore the possibility of developing new materials with superior properties by intentionally controlled decomposition of S-phase. To this end, a series of fast (50°C/min) annealing in a thermomechanical analyser and rapid (6000°C/min) annealing in a Gleeble machine were conducted at temperatures ranging from 425 to 640°C. XRD, GDS, SEM, TEM, hardness tests and electromechanical corrosion tests were used to characterise the microstructure and evaluate the properties of the rapidly annealed samples.

The results showed that the precipitation of nitrides and carbides from nitrogen and carbon S-phase started at 550°C and 650°C respectively during fast annealing (50°C/min); however, no precipitation was observed during rapid annealing (6000°C/min) of nitrogen and carbon S-phase up to 590°C and 650°C respectively. Nitrogen and carbon diffused inward into the substrate during both fast and rapid annealing of nitrogen and carbon S-phase, thus leading to increased thickness of the nitrogen and carbon S-phase layers. The hardness of nitrogen and carbon S-phase can be effectively increased by fast annealing at 475°C and 625°C respectively most probably due to the formation of Cr-N and Cr-C clusters or GP zones. When annealed at the same temperature, a higher hardness can be produced by rapid annealing than by fast annealing. Taking hardness, corrosion resistance and layer thickness into account, the optimal annealing temperature for fast annealing to generate hard and corrosion resistant S-phase is 425°C for PN sample and 525°C for PC sample. Therefore, it is possible to further harden and improve the corrosion properties of S-phase by fast and rapid annealing, which could pave the way towards new materials with superior properties.

15:15 - 15:40 Linear scratch tests of active screen low temperature plasma nitrided Oral #13 Martensitic stainless steel Luis Armando Espitia Sanjuan 1*; Hanshan Dong 2; Xiaoying Li 2; Carlos E. Pinedo 3 and Andreas P. Tschiptschin ⁴ ¹ Engineering, Science and Technology Research Group, University of Córdoba, Montería, Colombia ² School of Metallurgy and Materials. The University of Birmingham, Birmingham B15 2TT, UK ³ University of Mogi das Cruzes, São Paulo, SP, 05305-000, Brazil ⁴ Metallurgical and Materials Engineering Department, University of São Paulo, São Paulo, SP, Brazil * luisespitia@correo.unicordoba.edu.co A nitrided case composed of expanded martensite and small quantities of hexagonal ε-Fe₂₄N₁₀ iron nitrides was formed in a martensitic stainless steel by means of active screen plasma nitriding process. Nanoindentation tests were carried out in order to assess the mechanical properties and to obtain an energy dissipation coefficient defined as the ratio of plastic to total deformation energy. Friction coefficient, failure mechanisms and critical load for cracking the nitrided case were determined using linear scratch test performed at both progressively-increased normal force and constant normal force according to ASTM C1624 standard. The scratch test results showed that the groove features and the friction coefficient could be well correlated to the energy dissipation coefficient. The expanded martensite strongly decreased the friction coefficient in comparison to the non-nitrided martensitic stainless steel. The critical load was 14N and tensile cracking was the failure mechanism acting in the nitrided case. 15:40 - 16:10 Coffee break 17:45 Meeting at symposium location and selected hotels for bus pickup 18:00 - 19:45 **DMME** lab tour 20:00 Mdina walking tour and symposium dinner (Da Luigi, Rabat)

Friday 24th June 2016 08:00 - 09:00 Registration

Morning Session | S-phase on Duplex and Martensitic stainless steel & Ni-based super alloys Session chair: Prof Marcel Somers

09:00 - 09:25 Oral #14

Surface hardening of duplex stainless steel by high-temperature solution nitriding with/without subsequent low temperature surface hardening

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Duplex stainless steels (DSS) are frequently used in highly demanding applications (e.g. offshore oil and gas) due to their high corrosion resistance. Unfortunately, the wear resistance of DSS is not optimal, which may pose a problem for various mechanical components such as pumps and valves. Corrosion and wear properties of DSS can be enhanced by interstitial alloying with, in particular, nitrogen. Nitrogen is already utilised as an alloying element in the duplex, super-duplex and super-austenitic stainless steels and has a documented strong positive influence on corrosion properties (e.g. PREN). Furthermore, nitrogen is a highly effective solid solution strengthener. However, normal steel making practice does not permit high nitrogen contents due to low nitrogen solubility in the melt and nitrogen loss during solidification; especially in the case of ferritic solidification, as is the case in DSS. Hence, even if substantial nitrogen contents are introduced in the melt, e.g. during high-pressure secondary metallurgy, the lower solubility in δ ferrite will lead to significant nitrogen losses, pore formation and possibly nitride formation during solidification. Alternatively, nitrogen can be introduced in the solid state through thermochemical surface engineering. Recent progress in development of gaseous surface engineering methods, where nitrogen is introduced in the solid state at low temperatures (<450°C) or at high temperatures (>1050°C), enables nitrogen contents ranging from 0.2 wt% to 10 wt%; with absence of nitride formation, which is a prerequisite for successful interstitial alloying with nitrogen. At temperatures above 1050°C nitrides are not stable and at temperatures below 450°C there is a substantial incubation time for nitride precipitation.

This research mainly deals with the high-temperature solution nitriding (HTSN) route to obtain high nitrogen contents in the surface and surface-near region of DSS. HTSN of DSS leads to formation of a fully austenitic case with high wear and corrosion resistance on a high strength ferritic-austenitic duplex core. The maximum attainable nitrogen content in the austenitic nitrogen-rich case is contingent on the nitrogen solubility limit in austenite, which increases with increasing temperature. However, the ferrite fraction in the duplex core also increases with temperature. Therefore, optimum nitriding temperature for HTSN of DSS is in the range of ~1150-1175°C. Case-depths from 0.5 to 2.0 mm and nitrogen contents in solid solution at the surface from 0.5–1.0 wt% N can be obtained. The HTSN treatment is an excellent "pre-treatment" for subsequent low temperature surface hardening by carburizing, nitriding or nitrocarburizing. The combined treatment leads to unsurpassed (localized) corrosion performance.

09:25 - 09:50 Oral #15

Surface co-alloying of corrosion resistant CM247LC superalloy with V/Ag & N

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Nickel based super alloys have good oxidation and creep resistance, which make them a desirable material for gas turbine engines and hot forming tools. The ever-increasing demand for severe service conditions and lifespan of components in turbine engines and hot forming tools necessitate for multi-functional surface properties in terms of high hardness, high wear resistance and low friction.

Therefore, in this work an innovative active screen plasma technology was used to co-alloy the surfaces of CM247 superalloy with both interstitial element (i.e. N) and substitutional alloying elements (such as V and/or Ag) to provide a synergy effect to enhance its hardness and tribological properties. Nitrogen can be used to effectively harden the surface; vanadium has potential to reduce the surface friction at high temperature while silver is deemed to act at a relatively low temperature.

Microhardness tests were conducted to evaluate the hardening effect and friction and wear tests at temperatures ranging from room temperature to 600°C were used to assess the tribological properties of the active-screen plasma modified surfaces. The experimental results have demonstrated that the active-screen plasma co-alloying can effectively harden the surface of CM247 superalloy. Nitrided layers with vanadium additive can significantly reduce material transfer and adhesive wear/galling. Nitrided layers with Ag/V additive can lower the friction coefficient of CM247 superalloy at a relatively low temperature.

09:50 - 10:15 Oral #16

Application of next-generation stainless steel and titanium surface hardening

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Anti-wear, anti-galling and scratch resistance are well know properties associated with low-temperature interstitial hardening processes, however corrosion resistance is not always unaffected, although claimed so. The surface hardening engineering company Expanite has developed a series of advanced processes for low temperature surface hardening of stainless steel and Titanium. The gaseous processes allow precise control for accurate tailoring of materials' properties including superior corrosion resistance.

Expanite processes are based on the founders' dedicated research in the field during the past 15 years. In particular, through extensive characterization of expanded austenite, analysis of thermochemical reactions on surfaces and development of dedicated furnace equipment, the hardening technology is matured and Expanite has since early work in 2010 established themselves with hardening centers in Denmark, US and Germany.

The present contribution gives an overview of some of the fundamental scientific aspects of low temperature thermochemical treatment of stainless steel and a unique insight into the applicability and the industrialization of Expanite processes. Selected technological examples of thermochemical treatment of stainless steel and Titanium are presented.

10:15 - 10:45

Coffee break

10:45 - 11:10

Oral #17

PIRAC nitriding of titanium alloys

<u>Glenn Cassar</u> ^{1*}; Bonnie Attard ¹; Adrian Leyland ²; Allan Matthews ²; Elazar Y. Gutmanas ³ and Irena Gotman ³

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Despite the popularity of a number of techniques for nitriding titanium, including gas and plasma nitriding, in many cases these processes may not be economically viable options for industrial applications particularly for 'real' complex components. This work focuses on the application of a relatively inexpensive treatment which is capable of remarkable improvement in the surface characteristics of titanium alloys; Powder Immersion Reaction Assisted Coating (PIRAC). PIRAC was applied to the ubiquitous Ti-6Al-4V and the high-performance near- α titanium Timetal 834. The behaviour of Timetal 834 following PIRAC treatment was markedly different when compared to Ti-6Al-4V; although for both materials the surface still hardened considerably and both alloys' tribological performance in dry sliding conditions improved compared to the untreated alloys.

11:10 - 11:35 Oral #18

Gaseous surface hardening of titanium and titanium alloys

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Titanium is a light-weight material characterized by excellent corrosion resistance- and biocompatibility and is the material of choice in a plethora of different applications and in many industries, e.g. for implants, in aerospace, chemical processing etc. Unfortunately, titanium suffers from poor wear resistance which makes it unsuited for applications where wear is encountered. A remedy for this inherent short-coming can be found in surface engineering. The 'classical' surface hardening route for titanium entails the use of nitrogen where the surface is converted into TiN/Ti2N nitrides with an underlying diffusion zone by gaseous nitriding in N2 gas at high temperature.

The first part of the present contribution addresses gaseous surface hardening of titanium by incorporation of (interstitial) oxygen, i.e. different oxidation processes. It will be shown that relative deep and hard diffusion zones of oxygen in solid solution can be obtained. The second part presents new routes for gaseous surface hardening of titanium; in particular emphasis is given to so-called mixed interstitial solid solutions and compounds in the Tibased system. It will be showcased that surface hardness values in the range 2000 to 2500HV are obtainable and case depths up to 1 mm can be achieved. Gaseous processing allows accurate control of the developing microstructural features and offers the possibility for tailoring the interstitial contents in the surface engineered layer/case.

11:35 - 12:00 Oral #19

Effect of oxidation temperature and surface roughness on tribological behaviour of thermally oxidized pure zirconium

Abdulkarim Alansari 1* and Yong Sun 1

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Thermal oxidation (TO) is an effective surface engineering technique to harden the surfaces of zirconium (Zr) and its alloys for improvement in friction and wear performance. Unlike TO of titanium where the rutile oxide layer formed tends to be fragile and flake off easily when it is thicker than about 2 microns, TO of zirconium can produce a thick and adherent ZrO₂ layer without the danger of flaking off. However, an excessively thick ceramic ZrO₂ layer can lead to embrittlement of Zr surface and deteriorate its tribological properties. In the present investigation, the effect of TO temperature and surface roughness on the tribological properties of pure Zr was investigated. TO was carried out at temperatures between 550°C and 700°C to achieve ZrO₂ layer thickness ranging from 2 um to 9 um. Surface roughness before TO was varied between 0.12 um (Ra) and 0.26 um (Ra). Tribological tests under unlubricated sliding conditions demonstrated that TO was effective in reducing friction and wear rate of Zr and once the ZrO₂ layer maintained its integrity with the substrate, TO temperature had no significant effects on friction and wear. However, under high contact loads, the ZrO₂ layer tended to suffer from cracking in the wear track. The thinner layer produced at 550°C suffered from cracking at a small load. Although increasing ZrO₂ layer thickness helped to increase load bearing capacity, cracking was unavoidable at high contact loads. It was also found that the cracks were confined to the very shallow surface of the ZrO₂ layer and did not penetrate to the layer-substrate interface. Roughening the surface before oxidation helped to further reduce friction at the early stage of sliding and more importantly to reduce the tendency of the oxide layer towards cracking during sliding.

12:00 - 12:25 Oral #20

Phase transformations during low temperature nitrided Inconel 718 superalloy

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The effects of the salt bath nitriding parameters on the microstructure, microhardness and corrosion behaviour of Inconel 718 superalloy at temperature ranging from 425-500 °C were investigated by X-Ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and corrosion test. Experimental results indicated that the microstructure and phase constituents of the surface nitridization are highly process dependent. When Inconel 718 superalloy was subjected to salt bath nitriding, the predominant phases of the nitrided layer were identified as expanded austenite (S phase), austenite and CrN. The thickness of the nitrided layer increased with the time of nitridization. Meanwhile, salt bath nitriding improved the surface hardness dramatically. The maximum value of hardness measured from the treated surface was 2100 HV0.1 after 16 h at 500 °C, which is about 5 times as hard as the untreated material (420 HV0.05). Proper low temperature nitriding can improve the erosion corrosion resistance. The sample that was nitrided for 4 h at 475 °C had the best corrosion resistance.

12:25 - 13:00

Closing session chaired by Prof Marcel Somers

13:00 - 14:30

Lunch break