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DAMAGE OF METAL SURFACES
BY A
HOT, DENSE DEUTERIUM PLASMA

Nicholas Frank Panayotou

1978

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BY A
HOT, DENSE DEUTERIUM PLASMA

Nicholas Frank Panayotou

Submitted in partial fulfillment
of the requirements for
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ABSTRACT

DAMAGE OF METAL SURFACES BY A HOT, DENSE DEUTERIUM PLASMA

NICHOLAS FRANK PANAYOTOU

In this thesis we have determined the effect of differences in alloy chemistry and systematic changes in microstructural interface density on the occurrence of unipolar arcing.

Unipolar arcing, a plasma-materials effect, is known to be a potent source of plasma impurities that has been observed in tokamak plasma experiments and it may also occur in anticipated Fusion Power Reactors. A high energy density, deuterium plasma device, the Columbia Electromagnetic Shock Tube provides plasma-wall conditions wherein arcing occurs. The plasma is characterized by an ion temperature, $T_i \sim 10^6$ °K and a particle density of $\sim 10^{16}$ cm⁻³.

Unipolar arcing is thought to depend on the ability of materials to sustain thermionic electron emission, as well as, the preferential emission of electrons due to the presence of both low work function impurities, and interfaces. The effect of thermionic emission is examined by comparing the refractory metals W, Nb, and Nb-751 alloy to the non-refractory stainless steels, AISI 304, 316 and Nimonic* PE-16. The effect of alloying additions is examined by

* Nimonic is a registered trademark of the H. Wiggins Co. Ltd., Hereford, U.K., a subsidiary of the International Nickel Co., USA.

comparing Mo containing 316 and Mo free 304 stainless steels as well as Zr containing Nb-751 and Zr free Nb. The effect of interface density is examined by systematically varying the density of microstructural interfaces in AISI 4130, a ferritic steel. Although most of the metals and alloys studied are candidate fusion reactor materials, 4130 was chosen on the basis of our ability to modify the density of microstructural interfaces in the material by heat treatment.

Unipolar arcing is characterized by the formation of cathode spots on material surfaces. The damage sustained by material surfaces is analyzed by determining the total area coverage due to cathode spot remnants, as a function of plasma exposure or fluence. The influence of existing cathode spot remnants on further unipolar arc damage is analyzed by determining the mean area, density and morphology of cathode spot remnants as a function of fluence.

We have determined, for example, that unipolar arcing occurs on all metals and alloys tested; that the surface area affected by unipolar arcing is surprisingly similar even for different metal systems; that the effect of some alloying additions on unipolar arcing is minor, and that arcing is very sensitive to microstructural interface density for non-refractory metals.

The results are discussed with respect to available unipolar arcing theory. The significance of the work, in terms of actual power reactors is assessed and the need for further work defined.

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1. INTRODUCTION

It is very well known that high atomic weight (i.e. high Z) plasma contamination may be a serious problem for fusion power development.¹⁻⁶ Tokamak designs employing a divertor to remove impurities at the plasma edge are now being built and studied, but a divertor seriously complicates a fusion reactor design. It is important to understand the source mechanism of high Z impurities in Tokamak plasmas. It appears that a major source of plasma impurities may be due to the erosion of material surfaces by unipolar arcs. Unipolar arcing, a plasma-material effect, was first observed in 1959.⁷⁻⁹ Material damage, due to unipolar arcing, has not been systematically studied previously.

Unipolar arcing requires that a metallic surface in the proximity of a plasma attain a net negative potential of the order of 10 volts with respect to the plasma, and that the ion current to the surface exceed about 10 amps.⁷ Localized sources of electrons or cathode spots can then form at preferential sites on the metallic surface.¹⁰ Cathode spots can be characterized by high current densities⁷, of about 10^6 amps $\cdot$ cm⁻² and high erosion rates⁷, of about 10^{-4} gm $\cdot$ coulomb⁻¹ or about 10^{24} atoms per sec per cm², and these intensities can cause localized melting.⁷⁻¹⁷ In unipolar arcing, the same surface acts as both the anode and cathode of the arc discharge, since the arc circuit in that case involves the emission of electrons from the cathode spots and a return flow of plasma electrons to the rest of the surface.

Initial studies of unipolar arcing were undertaken by various groups at the United Kingdom Atomic Energy Research Establishment.¹⁸⁻²¹ These studies were limited in part by the relatively low temperature (i.e., about 100 eV) plasmas then available.⁷ Only apparent arcing rates were determined (number of arcs per number of device discharges) and the morphology and the extent of damage due to cathode spots were not reported.

Cathode spots also occur in devices which involve the formation of an arc between two distinct electrode surfaces. Such bipolar arcing is of practical interest to devices such as vacuum switches and rectifiers.^{11,12,22-24}

It appears that material parameters may influence the severity and number of cathode spots which form. The ability of a cathode spot to erode the material on which it forms depends upon the chemistry of the material.²⁵ Materials such as stainless steels, which cannot sustain significant thermionic emission, form only erosive cathode spots. Thermionic materials, such as the refractory metals W and Nb are known to sustain incandescent, as well as erosive cathode spots.^{13,26,27} Incandescent cathode spots are characterized by low current densities of about $500 \text{ amps} \cdot \text{cm}^{-2}$ and negligible erosion rates.¹³ The relative extent, though of erosive damage from cathode spots on refractory versus non-refractory metals has not been determined.

Localized emission of electrons, has been found to precede the formation of arc discharges.^{14,28-35} The enhanced emission is attributed to the presence of compositional and/or structural

heterogenities at the surface of a metal or alloy. Specifically, low work function impurities are thought to enhance electron emission³⁶, and their presence should increase the number of potential sites for cathode spot formation. The effect of these minor variations in alloy chemistry on cathode spot damage has also not been determined. Structural discontinuities, on the other hand, provide for enhanced local electron emission through the high local electric fields associated with them.^{34,35,37-40} The structural discontinuities include interfaces such as grain boundaries^{14,37}, precipitate and inclusion-matrix interfaces^{18-21,29,30}, as well as interfaces due to surface films^{13,21,28}, dust particles³¹ and other surface contamination. The structural effect on cathode spot damage, although identified as an effect, has also not been systematically studied.

For this thesis we have used a high energy density plasma device, the Columbia University Electromagnetic Shock Tube, to study unipolar arc damage sustained by a range of metals and alloys. The study really began with the initial observation by Fineberg^{41,42} of several unipolar arc tracks in his plasma-wall heat transfer study using this shock tube.

In this thesis we seek to determine the dependence of unipolar arc damage on:

- i) major variations in alloy chemistry, by comparing niobium, Nb-751, and two forms of tungsten with the

stainless steels Nimonic* PE-16, and AISI 304 and 316,

- ii) minor variations in alloy chemistry by comparing molybdenum free AISI 304 and molybdenum containing AISI 316 stainless steels, and zirconium free niobium and zirconium containing Nb-751 alloy,
- iii) systematic changes in the density of microstructural interfaces, by comparing three heat treated forms of AISI 4130 steel, martensitic for high density, austenitized for low density and spheroidized for medium interface density,
- iv) the effect of interfaces is also determined by comparing polished versus metallographically etched PE-16.

* Nimonic is a registered trademark of the H. Wiggins Co. Ltd., Hereford, U.K..

2. EXPERIMENTAL PROCEDURE

The Columbia University Electromagnetic Plasma Shock Tube produces a hot ($T \sim 500 \text{ eV} \sim 6 \times 10^6 \text{ }^\circ\text{K}$) dense ($n \sim 10^{22} \text{ m}^{-3}$) plasma by shock wave heating and compression. Prior to the present materials research this shock tube was used to experimentally study the physics of very strong shock waves. The shock created plasma was studied extensively and its properties carefully measured.⁴³⁻⁴⁹

2.1 Principle of Shock Tube Operation

The plasma shock tube is shown schematically in Fig. 1. Both inner and outer conductors of the shock tube are made of oxygen free, high conductivity (OFHC) copper cylinders, 12.7 mm thick and approximately 1.8 m long. The plasma is produced in the 5 cm annulus between the 23 cm diameter outer and the 13 cm diameter inner cylinders. A copper rod, which is electrically insulated from both conductors except for a common ground, carries a bias current and is located on the axis of the cylinders.

The operation of the shock tube first involves evacuation of the coaxial tube to a pressure less than 10^{-4} Pa . The tube is then filled with deuterium gas to a pressure of 6.6 Pa. At this point a slow rise time capacitor bank ($C = 1200 \text{ } \mu\text{F}$, $t_{\frac{1}{4}} = 70 \text{ } \mu\text{s}$, $v = 7 \text{ kV}$) is discharged through the bias rod to produce a transverse magnetic field of 3.6 kG. When this transverse magnetic field is approximately constant, a fast capacitor bank ($C = 57 \text{ } \mu\text{F}$, $t_{\frac{1}{4}} = 3.5 \text{ } \mu\text{s}$, $v = 35 \text{ kV}$) is discharged between the inner and outer conductors, thus forming a

Fig. 1. Schematic of Columbia University Electromagnetic Plasma Shock Tube.

radial current sheet. This current interacts with the magnetic field resulting in an axial $\vec{J} \times \vec{B}$ force which propels the current sheet at high speeds (on the order of $3.5 \times 10^5 \text{ m} \cdot \text{sec}^{-1}$) along the tube axis away from the launching wall and toward the reflecting end wall of the shock tube.

It is this magnetic piston that drives the shock wave, which in turn heats, ionizes and compresses the deuterium gas. The highly conductive plasma created by the shock wave builds up in front of the piston. At some distance, much shorter than the length of shock tube, the shock front fully separates from the drive current and between these two is a nearly current free plasma. The shock tube end wall is a flat, boron nitride wall that reflects the shock wave, which in turn heats and compresses the plasma further and brings it to rest. Each shot or pulse of the shock tube is followed by evacuating the device in preparation for the next shot. The average time between shots is approximately 10^2 sec.

Previous experimental research^{45-49,64-67} measured the actual plasma state in the section of the tube which is now the materials test section. This section constitutes the final 30 cm of the shock tube.

2.2 Summary of Plasma Conditions

The plasma temperature, $T = T_e + T_i$, where T_e and T_i are the electron and ion temperatures, respectively, is known to be a function of shock wave speed for a given value of initial deuterium

gas pressure and initial transverse magnetic field, see Fig. 2.⁴¹ The shock speed is monitored during an experiment by using magnetic field probes, the details of which can be found elsewhere.⁵⁰ For our operating conditions the shock speed was found to be $2.80 \cdot 10^5 \text{ m} \cdot \text{sec}^{-1}$, which corresponds very well to the measured plasma temperature of $5.8 \pm 1.2 \cdot 10^6 \text{ OK}$ (about $500 \pm 100 \text{ eV}$).⁴⁹ Since the electron temperature has been carefully measured and found to be only about 30 eV, then T is on the order of T_i .^{46,48} The energy initially deposited at the shock tube wall is due primarily to the ions which are within about one gyroradius of the wall.⁴¹ The gyroradius, r_g , is the radius of the helical trajectory described by an ion spiraling about a magnetic field line.

The characteristic interaction time for the initial energy deposition is the period which describes the rotation of the ions about the magnetic field lines, i.e. τ_g , the gyroperiod. The flux of ions, ϕ_i , to the specimen surface can be expressed by

$$\phi_i \approx \rho \cdot r_g \cdot \tau_g^{-1} \quad (1)$$

where ρ is the plasma density, which has been experimentally determined⁴¹ as $3 \cdot 10^{16} \text{ particles} \cdot \text{cm}^{-3}$. For our experiment the values of r_g and τ_g are about 1.0 cm and $4 \cdot 10^{-7} \text{ sec}$. The ion flux to specimen surfaces is, on the order $10^{23} \text{ ions} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$. Furthermore the power deposition, or plasma wall loading sustained by specimen surface is given by $\phi_i \cdot T_i$, and is $10^7 \text{ watts} \cdot \text{cm}^{-2}$.

Fig. 2. Calculated dependence of plasma temperature on shock wave speed for the deuterium fill pressure and magnetic field used.

The heat transfer to the shock tube wall for times greater than a single gyroperiod has been carefully measured and analyzed.^{41,45} The rate of energy deposition to the wall though, is greatest during that first gyroperiod. It is suspected that the major material damage observed is due mainly to the energy transfer which occurs during the first gyroperiod.

2.3 Comparison of the Shock Tube Plasma-Wall Conditions with Fusion Power Reactor Plasma Conditions

Table 1 compares the shock tube plasma conditions to those anticipated at the first wall¹ and at the limiter⁵¹ of a tokamak power reactor. The energy of the deuterium ions produced in the shock tube is of the order of the energy of the deuterium plasma ions anticipated near the first wall of a fusion reactor, but it is somewhat lower than the ion energies expected at the limiter. The importance of plasma-wall interactions involving < 1 keV deuterium ions is just beginning to be generally appreciated.⁵² The more energetic (23 keV) deuterium ions in the interior of the plasma are not expected to have as strong an interaction with the first wall as the lower energy deuterium ions closer to the wall. Structures, such as limiters, beam dumps and armor plate are expected to be subjected to bombardment by these higher energy particles.

In terms of particle flux and plasma wall loading, the shock tube plasma conditions appear comparable to the plasma conditions anticipated at the limiter of a power reactor but are greatly in excess of the conditions at the first wall. The basis of this large divergence in the wall loading parameter is due to the difference in

TABLE 1

Comparison of the shock tube plasma conditions and two plasma conditions in a tokamak power reactor.

<u>ITEM</u>	<u>SHOCK TUBE</u>	<u>TOKAMAK WALL</u> ¹	<u>TOKAMAK LIMITER</u> ⁵¹
Energy Per Particle (J)	10^{-16}	10^{-16}	10^{-14}
Particle Flux ($\text{cm}^{-2} \cdot \text{sec}^{-1}$)	10^{23}	10^{13}	10^{21}
Plasma Wall Loading (watts $\cdot \text{cm}^{-2}$)	10^7	10^{-3}	10^7
Duty Cycle (sec $\cdot \text{cycle}^{-1}$)	10^{-7}	10^4	10^4
Duty Cycle-Particle Fluence ($\text{cm}^{-2} \cdot \text{cycle}^{-1}$)	10^{16}	10^{17}	10^{25}
Duty Cycle-Plasma Energy Fluence (J $\cdot \text{cm}^{-2} \cdot \text{cycle}^{-1}$)	10^0	10^1	10^{12}

the characteristic operating times or duty cycles of the shock tube and of a tokamak power reactor. The use of a parameter such as wall loading implies that the energy deposition per unit area, normalized with respect to time will be an important design characteristic. This is valid for any steady state device in that the value of the wall loading parameter will, say, in the case of the neutron loading of a fuel cladding in a fission reactor, determine the operating temperature of the cladding. For a pulsed device though, such as the shock tube, the operating temperature of the specimen is controlled not only by the wall loading but is also dependent upon the repetition frequency of the energy deposition. Since the minimum time between shock tube shots is $\sim 10^2$ sec, while the energy deposition occurs over $\sim 10^{-7}$ sec, the energy which is deposited during each pulse is largely dissipated before the next pulse can even occur. Therefore, while the normalized energy deposition rate, i.e., wall loading on a per second basis, is clearly valid for comparing the relative effects of different energetic fluxes in the same or similar (with respect to characteristic operating time) devices, it may not be a valid parameter to describe all material effects for devices whose operating cycles differ markedly. Table 1, therefore, also includes a comparison of the shock tube and a tokamak power reactor on a characteristic operating time or duty cycle basis.

This comparison shows that the particle and energy fluence obtained in the shock tube are comparable to the conditions at the first wall but not at the limiter of a power reactor. This implies that by repeated pulsing of the shock tube we can simulate the interaction of deuterium particles and the first wall of a tokamak

power reactor in terms of energy and particle deposition. In fact, a plasma shock tube exposure (1000 pulses), which can be accomplished in approximately 20 hours, is equivalent, in terms of deuterium particle and energy fluence, to the deuterium exposure which would occur during approximately one year of tokamak power reactor operation. However, the devices are clearly different in that the rate of energy deposition, i.e. power, is not comparable so that, while the tokamak process may be thought of as isothermal, the shock tube involves an adiabatic-heating type process. The shock tube can, therefore, simulate the deuterium particle and energy fluence anticipated at the tokamak first wall and this simulation will be applicable to any plasma-material effect which is independent of the rate of particle (and therefore energy) deposition.

2.4 Design and Construction of a Materials Test

Section for the Plasma Shock Tube

The design of the test section was determined by the following requirement. Specimens to be exposed had to be accessible during a test so that they could be removed and replaced for the determination of microstructural damage without breaking the shock tube's vacuum or disturbing the other specimens. The alterations had to be compatible with the operation of the device, and for good design, previously existing ports, were utilized where possible to minimize the number of potential new leak sites.

This requirement was met by using a rod type specimen shown in Fig. 3. The use of these cylindrical specimens (6.05 mm

material: as specified

Rod Type Specimen

Specimen Holder (material : brass)

Fig. 3. Test specimen and holder.

in diameter) and specimen holders illustrated in Fig. 3 and the valve assembly shown in Fig. 4 provided specimen accessibility during testing. The removal of these specimens from the shock tube environment necessitates only the withdrawal of the specimen-holding assembly until the exposed surface is at point A (Fig. 4), allowing the ball valve to be rotated by a one-quarter turn, thus sealing the shock tube. Note that although these valves were not designated by the manufacturer as vacuum valves, we found that they could be modified as shown in Fig. 4. Since it was found that the inside diameter of the valve could not be enlarged without causing the valve to leak, the specimen-holder assembly was designed with the maximum allowable diameter specified so that it would pass, without interference, through the 6.35 mm (0.250 in.) ball valve. Special attention was given to the alignment of the fittings and the valve bore (Fig. 4).

Accurate positioning of the surface of the cylindrical specimens is critical to the test and was accomplished by careful alignment of the first set of eight specimens while the shock tube was open to atmosphere. The adjustable collars shown in Fig. 3 were then set so that this position would be maintained during a test. The correct alignment of all subsequent specimens was easily obtained by carefully comparing new (to be inserted) and exposed (just removed) specimen assemblies, and setting the adjustable collar so that the proper specimen surface-to-collar distance was duplicated.

The eight valve assemblies and four large 5.08 cm diameter ports are positioned symmetrically about the circumference of the shock

Fig. 4. Vacuum valve assembly which allows for easy access to the shock tube environment.

Fig. 5a. Detail of materials test section.

Fig. 5b. Pictorial view of materials test section in place on the shock tube.

tube, and are located at 4.45 cm and 14.60 cm, respectively, away from the boron nitride reflecting wall. The large diameter ports were utilized to expose reduced section mechanical test specimens to the plasma. Both sets of ports are in the area of the shock tube in which the plasma properties have been well studied.⁴⁶

The construction of the materials test section utilized the existing shock tube section which was equipped with a multitude of ports and had been used mainly for plasma physics studies. The fully assembled materials test section can be seen in Fig. 5b. Although we were able to utilize some of the ports already in place, our design required machining four new ports for the valve assemblies and two new large diameter ports. Vacuum valve assemblies connected to the shock tube via previously existing ports required the swagelock-to-one-quarter inch pipe reduction fitting shown in Fig. 4. Where new ports were made, a 6.35 mm diameter hole was drilled and counterbored so that a one-quarter inch pipe nipple could be used. The valve assembly could then be threaded directly to this nipple. All threads were wrapped with Teflon* tape prior to assembly. The large diameter ports were milled with a 5.08 cm diameter hole, and a flat where the O-ring, located on the specimen holder, could seat properly and the drilling and tapping of four holes for the 8-32 flange alignment studs.

The edges of all samples are positioned flush with the shock tube wall but the specimens were not contoured to exactly match the curvature of the tube. However, due to the comparatively large size

* Teflon is a registered trademark of the E.I. duPont de Nemours & Co. Inc.

of the shock tube and the small specimen size, the maximum deviation from the curvature of the shock tube is approximately 0.2%.

Assembly of the shock tube with the materials test section was followed by a careful check of the vacuum system and a thorough leak testing of all fittings. We studied different samples, by metallographic observation, to learn whether the damage sustained by material specimens in different rod ports was a function of the azimuthal position of the sample. No significant difference has been found to date.

2.5 Specimen Preparation and Material Description

The metals and alloys used for this study are listed in Table 2. The details of the surface preparation procedure used for each material are given in Appendix I. Most specimens were machined from commercial grade rod or sheet stock. The tungsten specimens were cut from the stock material by electric discharge machining prior to final machining. Metallographic analysis of the tungsten specimens prior to exposure ensured that only specimens with approximately equiaxed grain structure were used for testing. All specimens were initially prepared to a metallographically prepared, lightly etched condition where interfaces stand out in relief. A series of PE-16 specimens were, however, repolished using 0.05 μm alumina abrasive, until all the remnants of etching were removed. This is defined as the polished surface condition.

The nominal composition, prior history, average grain diameter and inclusion content of the metals and alloys used in this

TABLE 2

Material Specifications

<u>Material</u>	<u>Composition (wt %)</u>	<u>Prior Treatment</u>	<u>Inclusion, precipitate content (area %)</u>	<u>Mean Grain Diameter (μm)</u>
AISI 304	10 Ni, 19 Cr, bal Fe	annealed, air cooled	none	25
AISI 316	12 Ni, 17 Cr, 2.5 Mo (1.47 at % Mo) bal. Fe	annealed, air cooled	none	25
Nimonic PE-16	43 Ni, 16 Cr, 1.25 Al, 3.25 Mo, 0.25 Co, 0.5 C, 0.26 Si, 0.035 Zr, 1.0 Mn, 1.25 Ti, bal Fe	age hardened 0.5 hr at 1040°C 12 hrs at 700°C	TiC 1%* Ni ₃ (Al,Ti)	20
AISI 4130	(in at %) 1.18 Mo, 1.05 Cr, 0.50 Si, 0.52 Mn, 1.40 C bal. Fe	i) 0.5 hr at 843°C, oil quench, 2 hrs at 250°C ii) 0.5 hr at 843°C, furnace cool iii) 0.5 hr at 843°C, furnace cool, 150 hrs at 677°C	Mn S, 1%*	20

.....continued on next page

* As determined by energy dispersive x-ray analysis.

TABLE 2
(continued)

<u>Material</u>	<u>Composition (wt %)</u>	<u>Prior Treatment</u>	<u>Inclusion, precipitate content (area %)</u>	<u>Mean Grain Diameter (μm)</u>
Arc Cast W	(in ppm) 30 Al, 60 Ca, 10 Cu, 25 Fe, 2 Mg, 50 C, 1.1 H, 7 N, 27 O	arc cast	carbides, 1%	20
Pressed and Sintered W	(in ppm) 10 As, 30 Al, 15 Ca, 10 Cr, 10 Cu, 20 Fe, 10 Mg, 10 Mn, 50 Mo, 20 Ni, 10 Si, 50 C, 5 H, 10 N, 100 O	pressed and sintered	carbides, 1%	50 (+80 -20)
Nb	99.8 Nb	annealed	none	10
Nb-751	(at %) 1.02 Zr, 0.05 Ta, bal. Nb	annealed	none	10

study are also listed in Table 2. Nimonic PE-16, a precipitation strengthened austenitic stainless steel, consists of fcc grains and is strengthened by the addition of Ti and Al to form the low volume fraction of γ' ($\text{Ni}_3(\text{Al}, \text{Ti})$) precipitates. The pre-exposure microstructure of PE-16, in both lightly etched and polished surface conditions is shown in Fig. 6.

The stainless steels 304 and 316 are characterized by twinned fcc grains and exhibit comparable mean grain diameters. Typical pre-exposure micrographs of annealed 304 and 316 are shown in Fig. 7. The stainless steels 304 and 316 are distinguished mainly by the presence of 1.47 atomic percent Mo in 316. Mo has a slightly lower work function than the other constituents of 316 stainless.⁵³

One form of W, and both Nb and Zr containing Nb-751, were fabricated by vacuum arc casting. The Nb and Nb-751 were found to be relatively free of inclusions. The pre-exposure microstructure of these metals and alloys is shown in Figs. 8 and 9. Zr has a slightly higher work function than Nb.⁵³ The arc cast W was used in the as cast condition while both Nb and Nb-751 were in the annealed condition. The pressed and sintered W was found to have a larger mean grain diameter that varied quite widely. The fine structure, apparent in the pre-exposure micrograph of the pressed and sintered W, Fig. 9b, is due to the etching of subgrain boundaries.⁵⁴

The high strength low alloy steel 4130 was heat treated and tested in three distinct microstructural conditions: high interface density martensitic, low interface density austenitized and medium

(a)

(b)

Fig. 6. Pre-exposure differential interference contrast (DIC) photomicrograph of PE-16 in the etched (a) and in the polished (b) condition.

(a)

(b)

Fig. 7. Pre-exposure DIC photomicrograph of 304 (a) and 316 (b) stainless steel.

(a)

(b)

Fig. 8. Pre-exposure DIC photomicrograph of Nb (a) and Nb-751 alloy (b).

(a)

(b)

Fig. 9. Pre-exposure DIC photomicrograph of arc cast W (a) and pressed and sintered W (b).

interface density spheroidized. Figures 10 and 11 illustrate the pre-exposure microstructures of the three heat treatments. Oil quenching from 843°C and tempering for 2 hours at 250°C resulted in the formation of tempered martensite, Fig. 10. Furnace cooling from 843°C , after austenizing at that temperature, resulted in the formation of distinct colonies of coarse lamellar pearlite in a matrix of ferrite, Fig. 11a. The area fraction of pearlite is about 40 percent. The third heat treatment for 4130 involves holding at 677°C for 150 hours after an initial austenizing treatment. The result of this treatment is the spheroidizing of the pearlite lamellae and a partial homogenization of the microstructure. Note the presence of iron carbide particles within the ferrite grains, Fig. 11b, as compared to the relatively carbide free ferrite grains which resulted from the austenizing treatment, Fig. 11a.

2.6 Specimen Exposure and Analysis

Experience has shown that venting the shock tube to atmospheric pressure, even with argon gas present and for brief periods of time, contaminates the internal surfaces of the shock tube sufficiently to cause poor results when the vacuum is restored and the shock tube is first fired. These initial discharges produce a post shot gas pressure P_F , which is about a factor of 10 larger than the deuterium gas fill pressure, P_D . This high pressure results from desorption of gas molecules which accumulated on the shock tube walls. Thirty to fifty shots are then required to clean the tube. Specimens inserted into the shock tube during this cleaning procedure developed pronounced edge effects as shown in Fig. 12. Once the shock tube is clean, these

Fig. 10. Pre-exposure DIC photomicrograph of martensitic 4130 steel.

(a)

(b)

Fig. 11. Pre-exposure DIC photomicrograph of austenitized (a) and spheroidized (b) 4130 steel.

Fig. 12. Low magnification optical photomicrograph of a PE-16 specimen which was in place during the shock tube discharge cleaning procedure. Note the edge effects.

effects are not found, Fig. 13.

Specimens were subjected to, from 1 to 1000 plasma exposures. To obtain data after 100, 300, 600 or 1000 exposures, four separate specimens were employed. Low exposure data, i.e. after 1, 2, or 5 shots, were obtained for each material tested by analyzing the same specimen after each withdrawal from the shock tube environment.

Specimen analysis included the determination of the number of cathode spots using both optical and scanning electron microscope techniques, and the measurement of the remnant area coverage of the arcs. An energy dispersive x-ray analyzer was used in conjunction with the scanning electron microscope. Micro hardness data were also obtained, using a Leitz Miniload Microhardness Tester.

The determination of area coverage by cathode spot remnants involved the photomicrographic mapping of each specimen. The Normaski differential interference contrast technique⁵⁵ was used to produce some of the micrographs. This technique is particularly sensitive to changes in surface topography. Each exposed specimen was completely mapped at a magnification of about 40 diameters. The area of each cathode spot remnant which was resolvable at that magnification was measured. The spot remnants which were not resolvable were sampled by taking photomicrographs at about 300 diameters. As determined by carefully scanning specimen surfaces at higher magnification, the minimum cathode spot remnant diameter observed was 3 to 5 μm . For sampling purposes, therefore, magnifications in excess of 300 diameters were not required. Micrographs were taken in a systematic fashion--one at the

Fig. 13. Low magnification optical photomicrograph of a PE-16 specimen subjected to several plasma exposures after discharge cleaning procedure was completed. Note the lack of edge effects observed in Fig. 12.

center of the specimen and four more at positions north, south, east, and west of the center at a distance of about 0.25 cm from the center of the specimen. A cathode spot area distribution, that is, the total number of cathode spot remnants of each observed size, was obtained for the systematic sample. Typically, about 1% of the total specimen area was sampled, and 200 to 800 resolvable cathode spot remnants were observed depending on the number of plasma exposures. The validity of the systematic sample was checked by taking a second sample at random; again in areas of the specimen surface which, at 40 diameters, showed no resolvable cathode spot remnants. In all cases, the second nonsystematic sample was found to agree with the systematic sample to within one standard deviation of the mean of the area distribution.

Irrespective of magnification, the measurement of the area of a cathode spot remnant was accomplished by comparing the image of the remnant with a millimeter grid and then counting squares. This technique was calibrated by using the grid to measure the area of carefully drawn circles. Measured versus actual areas were plotted for the range of cathode spot remnant areas observed. The standard deviation of the data from a least-squares straight-line fit was determined as 0.115 (area units).

The total number of cathode spot remnants, N_t , is given by $N_t = N_{40} + N_{300} \times \frac{1}{A_s} \times (1 - A_{40})$ where N_{40} and N_{300} are the total number of remnants observed at 40 and 300 diameters respectively, A_s is the fraction of the total specimen surface area accounted for by the sample, and A_{40} is the area occupied by the remnants observed at 40 diameters, i.e., $(1 - A_{40})$ is that fraction of the specimen surface area over which

the sample, taken at 300 diameters, is assumed to be valid. The total area coverage, due to cathode spot remnants A_t , is therefore

$$A_t = \sum_i N_i A_i$$

where N_i is the number of cathode spot remnants which have an area of A_i . The mean cathode spot area was calculated as

$$\bar{A} = \frac{\sum_i N_i A_i}{N_t}$$

Copper droplets have been observed on exposed specimens. We believe that these droplets come from the walls of the shock tube itself, sourced by unipolar arcing. The droplets are no larger than 0.5 μm and they do not seem to be associated with the cathode spot remnants. A complication in this study was the observation of uniform coverage of exposed specimen surfaces by blister like features after about 600 plasma exposures or shots. These features appear to form as a film deposited on the specimen surfaces during a run. The film is composed mainly of boron (probably from the launching and reflecting end walls of the shock tube), silicon and carbon (vacuum grease, diffusion pump oil) and oxygen.⁵⁶

The interface density produced by heat treating 4130 steel was determined. A standard line intercept technique was used to determine the mean inter-lamellar spacing in pearlite and the mean inter-particle distance. The area fraction occupied by pearlite and by, say, the spheroidized carbide particles was determined by point counting.

3. EXPERIMENTAL RESULTS

A cathode spot remnant area distribution is shown in Fig. 14 for 1, 100, and 1000 plasma exposures of austenitized 4130 steel. The shape of the distribution is typical for all metals and alloys examined. The distribution shows that no incubation period is required for either the appearance of cathode spot remnants or the formation of large cathode spot remnants, say 100 times the area of the minimum cathode spot remnant observed. There is a trend towards the appearance of even larger cathode spot remnants, up to $5 \cdot 10^{-3} \text{ cm}^2$ after 1000 exposures as compared with only $2 \times 10^{-5} \text{ cm}^2$ after a single plasma exposure.

The remnant cathode spot area coverage, as a function of plasma exposure or fluence is shown in Figs. 15-17 for the stainless steels, the refractory metals and the three heat treatments of 4130 steel. The stainless steels, Fig. 15a, show area coverage of from 1.7 to 2.8 percent after 100 exposures and from 5.4 to 8.8 percent coverage after 1000 plasma exposures. In general 316 stainless has sustained the least area coverage and PE-16 (etched) the most.

The refractory metals, Fig. 15b, show area coverage which lies in a band ranging from 0.7 to 1.7 percent after 100 shots and from 2.2 to 3.4 percent after 1000 plasma exposures. This is about a factor of 2 less than the area coverage sustained by the stainless steels.

The area coverage for the three heat treatments of 4130 steel, Fig. 16, indicates somewhat different behavior. Although the austenitized

Fig. 14. Cathode spot remnant area distribution for austenitized 4130, after 1, 100 and 1000 plasma exposures. Note apparent separation of data as a function of exposure and the tendency for larger remnants after larger exposures.

Fig. 15. Percent area coverage due to cathode spot remnants as a function of plasma exposure for the stainless steels and the refractory metals and alloy. Note that the coverage even for these distinct metal systems is within a factor of 2.

Fig. 16. Percent area coverage due to cathode spot remnants as a function of plasma exposure for the high interface density martensitic, medium interface density spheroidized, and low interface density austenitized, conditions of 4130 steel. Note the rapid approach of the high interface density condition to saturation.

Fig. 17. Percent area coverage due to cathode spot remnants, as a function of the number of plasma exposures for high interface density martensitic, low interface density austenitized and medium interface density spheroidized conditions of 4130 steel.

condition sustained no more than 13 percent coverage after almost 1000 plasma exposures, the spheroidized condition has sustained almost complete coverage after almost 1000 shots, and the martensitic condition sustained more than 50 percent coverage after just 100 exposures, and complete coverage after only 600 exposures. Figure 17 shows the area coverage for 4130 steel after 1, 2 and 5 plasma exposures. Again the austenitized condition shows both the least coverage, about 1 percent after 5 shots, and the slowest increase in coverage as a function of exposure. The spheroidized condition shows almost 5 percent coverage after 5 shots and the martensitic condition has sustained over 20 percent coverage after only 5 shots or plasma exposures.

The mean contiguous cathode spot area, as a function of plasma fluence or exposure is shown in Figs. 18-20 for the stainless steels, the refractory metals and the three heat treatments of 4130 steel. Although the mean area for all the stainless steels after 100 exposures is comparable, Fig. 18a, the mean area for the polished condition of PE-16 is approximately constant over 1000 exposures while, the etched stainless steels show an increasing dependence of mean spot area on exposure. Etched PE-16 has the largest mean spot area, about $1.3 \cdot 10^{-5} \text{ cm}^2$, after 1000 exposures, followed by 304 and 316 stainless.

The refractory metals, Fig. 18b, show the opposite dependence of mean cathode spot area on exposure. The mean spot areas lie in a band which extends from about $5.5 \cdot 10^{-6} \text{ cm}^2$ to $9.5 \cdot 10^{-6} \text{ cm}^2$ after 100 exposures and from 4.8 to $1.0 \cdot 10^{-6} \text{ cm}^2$ after about 1000 exposures.

Fig. 18. Mean cathode spot remnant area as a function of plasma exposure for the refractory metals and stainless steels. Note the increasing dependence exhibited by the etched stainless steels as opposed to the decreasing dependence exhibited by the refractory metals.

Fig. 19. Mean cathode spot remnant area as a function of plasma exposure for 4130 steel.

Fig. 20. Mean cathode spot remnant area for the three conditions of 4130 steel, after 1, 2 and 5 plasma exposures. Note that the high interface density martensitic condition has the largest mean cathode spot remnant area, and that the remnant area of the low interface density austenitized condition is about constant with exposure.

Partial data for the mean contiguous spot size for 4130 steel is shown in Fig. 19. The data for the martensitic condition and for the spheroidized condition after about 300 exposures could not be obtained due to the large percent area coverage sustained by these specimens. The result for the austenitized condition shows a slightly increase in the mean spot area over 1000 shots, while the data for the spheroidized condition indicates a steep increase in mean spot size. Data for all three conditions of 4130 was obtained from low exposure specimens, Fig. 20. The martensitic condition shows the sharpest increase in mean spot area reaching about $8.5 \cdot 10^{-6} \text{ cm}^2$ after 5 exposures. The spheroidized condition shows a slower increase in mean spot size, reaching only about $1.2 \cdot 10^{-6} \text{ cm}^2$ after 5 exposures, and the mean spot area for the austenitized condition appears about constant over 5 exposures at about $1.7 \cdot 10^{-7} \text{ cm}^2$.

The density of contiguous cathode spots, as a function of plasma exposure is shown in Figs. 21-23 for the stainless steels, refractory metals and the three conditions of 4130 steel. The etched stainless steels, Fig. 21a, show a decreasing or approximately constant dependence of spot density on plasma exposure. The polished condition of PE-16 shows an increasing dependence, with the spot density exceeding $4.0 \cdot 10^5 \text{ spots} \cdot \text{cm}^{-2}$.

The refractory metals, Fig. 21b, generally show an increasing dependence of cathode spot density on exposure. Although the spot density after 100 exposures is comparable, the spot densities determined after 1000 exposures range from a low of about $6.0 \cdot 10^4 \text{ cm}^{-2}$

Fig. 21. Density of cathode spot remnants as a function of exposure for the stainless steels and refractory metals. Note the decreasing dependence exhibited by the etched stainless steels as compared to the increasing dependence exhibited by the refractory metals and the polished PE-16.

Fig. 22. Density of cathode spot remnants as a function of exposure for 4130 steel. Note the decreasing dependence indicated for the spheroidized condition and the increasing dependence exhibited by the austenitized condition after about 600 plasma exposures.

Fig. 23. Density of cathode spot remnants for 4130 steel, after 1, 2 and 5 plasma exposures. Note the high apparent spot density for the austenitized condition, which remains about constant with exposure, as compared to the low but increasing spot density for the spheroidized and martensitic conditions.

for pressed and sintered W, to a high of $3.0 \cdot 10^5 \text{ cm}^{-2}$ for Zr free Nb.

Available data for the dependence of contiguous cathode spot remnant density on plasma exposure for 4130 steel is shown in Fig. 22. Again the data for the martensitic condition and for the spheroidized condition after about 300 shots could not be obtained due to the complete coverage of the specimens. Still the data for the spheroidized condition indicates a decreasing dependence of spot density on plasma exposure, while the austenitized condition shows an initially constant spot density, of about $8.5 \cdot 10^4 \text{ cm}^{-2}$ which increases, after 600 shots towards a spot density of about $3.0 \cdot 10^5 \text{ cm}^{-2}$ after almost 1000 shots.

Low exposure, spot density data, is available for all three conditions of 4130 steel, Fig. 23. The austenitized condition shows the greatest cathode spot density and it is approximately constant over 5 plasma exposures at about $70 \cdot 10^3 \text{ cm}^{-2}$. Both the spheroidized and martensitic conditions show an increasing dependence of spot density on exposure which ranges from about 6 to $24 \cdot 10^3 \text{ cm}^{-2}$ after 100 exposure and from about 25 to $40 \cdot 10^3 \text{ cm}^{-2}$ after 1000 exposures.

A typical view of the damage sustained by the stainless steels, is shown in Fig. 24. Here etched PE-16 has been subjected to about 100 plasma exposures. Note the attack, as evidenced by the formation of craters at what was previously a cuboidal TiC inclusion, at grain boundaries, and at slip traces and grain interiors. Figure 25 shows an arc track found on etched PE-16 after about 100 plasma exposures. The track is about $160 \mu\text{m}$ in length, $3 \mu\text{m}$ wide and about $1 \mu\text{m}$ deep. Such

Fig. 24. Scanning electron microscope (SEM) photomicrograph of the surface of PE-16 (etched) after 125 plasma exposures ($1.25 \cdot 10^{18}$ ions $\cdot$ cm $^{-2}$). Note the loss of material at the TiC inclusion (center of view) and the selective attack at grain boundaries and slip traces.

Fig. 25. SEM photomicrograph of PE-16 (etched) after 125 plasma exposures ($1.25 \cdot 10^{18}$ ions $\cdot$ cm $^{-2}$). Note the arc track produced by unipolar arcing.

arc tracks have been found to be extremely rare perhaps because of the short time of each plasma exposure, but are illustrative of the loss of material sustained at a cathode spot site. This class of tracks have been observed on the walls of tokamak experiments.^{2,3}

Figure 26 compares the cathode spot remnants visible in the polished and in the etched conditions of PE-16. Although the remnant morphologies are similar after 100 exposures, they are distinct after about 600 exposures, Fig. 27. Large cathode spot remnants, shown in Fig. 27 and after 1000 exposures in Fig. 28, are characterized by extensive cracking, an apparent volume expansion and a microhardness of about three times as great as the bulk microhardness. These remnants were observed only on the etched stainless steels. Specimens were sectioned and metallographically prepared so that cross-sectional views through these large cathode spot remnants could be obtained, Fig. 29. These features involve cracking and a columnar structure which extends about one grain diameter into the bulk.

The morphologies of cathode spot remnants in Nb and Nb-751 are shown in Fig. 30 after 100 exposures and in Fig. 31 after 1000 exposures. The craters which are visible are indicative of unipolar arcing. Note the similarity in morphology of the spots in Nb as compared to those which occurred in Nb-751, and the lack of any apparent change in morphology even after 1000 exposures. The fine structure visible in Fig. 31 may be the result of the sputter etching of the lightly etched pre-exposure structure of Nb-751, Fig. 9b.

The morphological results for the two forms of W are shown

(a)

(b)

Fig. 26. DIC photomicrographs of polished (a) and etched (b) PE-16 after 100 plasma exposures (10^{18} ions $\cdot$ cm $^{-2}$). Note the similar morphology of the cathode spot remnants.

(a)

(b)

Fig. 27. DIC photomicrographs of polished (a) and etched (b) PE-16 after 600 plasma exposures ($6 \cdot 10^{18}$ ions $\cdot$ cm $^{-2}$). Note the difference in the morphology of the cathode spot remnants.

Fig. 28. SEM photomicrograph of the surface of etched PE-16 after 1000 plasma exposures (10^{19} ions $\cdot$ cm $^{-2}$). Note the large area occupied by the remnant. The cathode spot remnant is also characterized by a volume expansion and significant cracking.

Fig. 29. SEM photomicrographs of a cross-section through the remnant shown in Fig. 29. Note that the cracking extends about one grain diameter into the bulk. A columnar structure, distinct from the fcc structure of the bulk is also apparent.

(a)

(b)

Fig. 30. DIC photomicrographs of the surface of Nb (a) and Nb-751 (b) after 100 plasma exposures (10^{19} ions $\cdot$ cm $^{-2}$). Note the separate and distinct morphology of the observed cathode spot remnants.

(a)

(b)

Fig. 31. DIC photomicrographs of the surface of Nb (a) and Nb-751 (b) after 1000 plasma exposures (10^{19} ions cm^{-2}). Note that the observed cathode spot remnants are still separate and distinct. The grain boundaries of both specimens appear more distinct, probably due to sputter etching.

in Figs. 32 and 33. Again note the similarity in the morphology of the cathode spot remnants observed in either arc cast or pressed and sintered W, after 100 or even after 1000 shots.

The results for 4130 steel are shown in Figs. 34 and 35 after 100 plasma exposures. While cathode spot remnants observed on the austenitized condition of 4130 steel, Fig. 34a, are circular craters and melted areas with diameters of about 10-15 μm , some of the spots observed in the case of the spheroidized, Fig. 34b, and martensitic Fig. 35, conditions are large fern-like features 100 or 150 μm in size (maximum dimension). After 1000 exposures these large features have also appeared in the austenitic condition of 4130, Fig. 36a, while the spheroidized condition after 600 shots, Fig. 36b, illustrates the rapid approach to complete coverage. After 1000 plasma exposures, the martensitic condition of 4130, Fig. 37, seems to be only partially covered with cathode spot remnants. A detail of the central portion of Fig. 37a, though, indicates that this material has also undergone apparent melting and resolidification, Fig. 37b.

The interface densities resulting from the three heat treatments were determined. The austenitized condition consists of distinct high interface density colonies of pearlite separated by low interface density ferrite grains, Fig. 34a. The iron carbide (Fe_3C) inter-lamellar spacing is about 0.25 μm , resulting in a low overall interface density for the austenitized condition of about $3 \cdot 10^4 \text{ cm of interface} \cdot \text{cm}^{-2}$. The spheroidized condition, Fig. 34b, consists

(a)

(b)

Fig. 32. DIC photomicrographs of the surface of arc cast W (a) and pressed and sintered W (b) after 100 plasma exposures.

(a)

(b)

Fig. 33. DIC photomicrographs of the surface of arc cast W (a) and pressed and sintered W (b) after 1000 plasma exposures. Note that the cathode spot remnants are still separate and distinct.

(a)

(b)

Fig. 34. DIC photomicrographs of the surface of 4130 steel in the austenitized (a) and spheroidized (b) condition. Note that the cathode spot remnants are localized in the austenitized condition to the pearlite colonies while in the spheroidized condition the remnants are not restricted.

Fig. 35. DIC photomicrograph of the surface of martensitic 4130 after 100 plasma exposures (10^{18} ions $\cdot$ cm $^{-2}$). Note the large cathode spot remnants.

(a)

(b)

Fig. 36. DIC photomicrograph of the surface of austenitized 4130 after 1000 plasma exposures (10^{19} ions $\cdot$ cm $^{-2}$) (a) and spheroidized 4130 after 600 plasma exposures ($6 \cdot 10^{18}$ ions $\cdot$ cm $^{-2}$) (b). Note the appearance of some cathode spot remnants which are no longer restricted to pearlite colonies, on the austenitized condition. The remnants observed on the spheroidized condition are quite extensive, with few individual remnants resolvable.

(a)

(b)

Fig. 37. DIC photomicrograph of martensitic 4130 after 1000 plasma exposures (10^{19} ions $\cdot$ cm $^{-2}$). Although individual remnants appear resolvable (a), a detail of the surface structure next to a large remnant indicates that it has also undergone apparent melting.

of partially spheroidized pearlite colonies and a distribution, in the pro-eutectoid ferrite grains, of 0.5 μm diameter iron carbide precipitates having a mean inter-precipitate spacing of about 2.3 μm . To a first approximation the interface density for the spheroidized condition is approximately that of the austenitized condition plus the interface density of the 0.5 μm iron carbide precipitates, or a total of $3.5 \cdot 10^4 \text{ cm} \cdot \text{cm}^{-2}$. The martensitic condition, Fig. 35, is characterized by a network of 10 μm by 2.5 μm plates and by a fine distribution of ϵ -carbide. The heat treatment employed for the martensitic condition is known to produce a continuous network of ϵ -carbide precipitates on the subgrain boundaries of the martensite plates.⁵⁸ The sub-boundaries have an average spacing of about 10^{-5} cm ,⁵⁸ resulting in a total interface density for the martensitic condition of about $4.5 \cdot 10^5 \text{ cm} \cdot \text{cm}^{-2}$ or about 15 times greater than the austenitized condition. It is also known that large amounts of retained austenite should not be produced in 4130 by the heat treatment employed⁵⁹, and in fact none was observed.

4. DISCUSSION

In this study it is shown that systematic changes in alloy chemistry have a measurable but not large effect upon unipolar arcing, as determined by the area coverage due to cathode spot remnants. Unipolar arcing, however, appears very dependent upon alloy microstructure for some materials.

4.1 The Effect of Major Differences in Alloy Chemistry

Refractory metals due to their relatively high boiling points can be heated by positive ion bombardment to the temperatures required for thermionic emission. A simple estimate, assuming no heat transfer, shows that surface temperatures sufficient for thermionic emission can be attained in the shock tube. For example the energy deposited during the first gyroperiod, at the specimen surface, can heat W to a temperature of about 3000°C over a depth of about 1 μm . This estimate uses ambient temperature values for the heat capacity⁶⁰ of W. Using the Richardson equation, for thermionic electron emission²²

$$J = AT^2 \exp(-\phi/kT)$$

where J is the current density, A is a constant ($120 \text{ amp} \cdot \text{cm}^{-2} \text{ } ^\circ\text{K}^{-2}$), T is the absolute temperature and ϕ is the work function of the emitter (about 4.54 eV for W), we find that for a temperature of about 3000°C the thermionic current density from W will be about $10^2 - 10^3 \text{ amp} \cdot \text{cm}^{-2}$.

This current density, is sufficient for the formation of an incandescent cathode spot.¹³ In spite of their apparent ability to emit thermionically and thus possibly avoiding the formation of erosive cathode spots, the refractory metals and alloys have sustained erosive cathode spots. Furthermore, the extent of the erosive cathode spot remnants in terms of area coverage is comparable to the coverage sustained by the low boiling point non-refractory stainless steels.

Although the current density is sufficient for the formation of an incandescent spot, an analysis of the characteristic cooling time of a layer of W 1 μm thick indicates that it is about 10^{-8} sec. The characteristic cooling time τ , is given by $H_C \cdot l^2 \cdot K^{-1} \cdot \rho$ where ρ is the metal density, H_C is the heat capacity⁶¹, K is thermal conductivity⁶² and l is the characteristic cooling distance ($1 \cdot 10^{-4}$ cm). The major energy influx to the shock tube wall occurs over 10^{-7} sec. The occurrence of thermionic emission or the formation of nonerosive incandescent cathode spots may, therefore, be limited in the shock tube. This does not imply however, that our observation of erosive cathode spots is due only to the high energy density characteristic of the shock tube. Erosive cathode spots have been observed by others on refractory metals^{13,26,27}, and we have determined that the unipolar arc damage sustained by the refractory metals is significant under conditions where thermionic emission is not precluded.

The results for the refractory metals and alloys and the stainless steels do differ, however, in terms of the morphology of the arc remnants. Even after 1000 plasma exposures

(a fluence of 10^{19} ions $\cdot$ cm $^{-2}$) the remnants observed on the refractory metals and alloys, Figs. 31 and 33 are separate and distinct. The decrease in mean area and increase in density of cathode spot remnants for the refractory metals and alloys indicates no spot growth, or more properly no tendency to form contiguous cathode spot remnants.

The remnants observed on the stainless steels, Figs. 26-28, on the other hand, do show a tendency to form contiguous cathode spot remnants. This is reflected by the mean area and density data, Figs. 18 and 21.

The formation of contiguous cathode spot remnants on stainless steels but not on the refractory metals and alloys is not due to the relative size of the cathode spots formed in these distinct metal systems. Figure 18 shows that the mean spot remnant area after 100 plasma exposures (10^{18} ions $\cdot$ cm $^{-2}$) is comparable for both metal systems. Furthermore, despite the differences in both microstructure and composition, of arc cast and pressed and sintered W, Fig. 9, Table 2, the area coverage due to cathode spot remnants, Fig. 15, and the mean area, density and morphology of the cathode spot remnants as a function of plasma exposure or fluence, Figs. 18, 21, 32 and 33, are surprisingly comparable. It appears therefore that unipolar arcing at least for the refractory metals is not strongly dependent on microstructure. This is not the case, however, for the non-refractory metals and alloys.

The fact that we are able to reverse the dependence of mean

area and density of cathode spot remnants just by polishing away the etched microstructure of PE-16 indicates that the formation of contiguous remnants on non-refractory alloys, like the stainless steels might be strongly dependent upon microstructure. Unipolar arcing on etched stainless steels appears to seek out certain sites, leading, as mentioned above to the formation of contiguous cathode spot remnants. The morphology of the large cathode spot remnants, which are observed, Figs. 27a-29, may be due to a phase transformation. The columnar structure visible in a cross-section, Fig. 29, may not be due solely to melting and rapid resolidification. Specimens of PE-16 were subjected to irradiation by a TEA laser⁶³ having a power level comparable to the shock tube, to determine if the structure observed in Fig. 29 is indeed a resolidification structure. The results are shown in Fig. 38. The cross-sectional view of the specimen shows evidence of melting, but there is no evidence of the columnar structure produced in plasma exposed specimens. This suggests that the structure might be due to the presence of deuterium and could be that of nickel β hydride, a brittle intermetallic compound routinely produced during the electrolytic charging of nickel containing ferrous alloys with hydrogen.⁶⁴ The deuterium which is required for the transformation, is available both from the plasma ion flux to the specimen surface (about 10^{16} ions $\cdot$ cm⁻² $\cdot$ exposure⁻¹) and from the unipolar arc current.

It is estimated that about 10 percent of the current density of the cathode spots is accounted for by positive ions moving from the plasma to the spot.⁶⁵ This represents an additional deuterium ion flux

Fig. 38. SEM photomicrographs of PE-16 subjected to increasing numbers of laser pulses. The exposed surface is at the left in (a). The power level of the laser is comparable to that of the shock tube. Although the surface has certainly melted, there is no sign (d), of the columnar structure observed in shock tube exposed specimens of PE-16, Fig. 30.

to the cathode spot sites of $5 \cdot 10^{23}$ ions $\cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$, or about a factor of 5 more than the incident flux due to plasma bombardment. It is estimated that this total deuterium flux is sufficient to produce β hydride. The added deuterium influx due to unipolar arcing could explain why the apparently transformed structure is localized and has not formed uniformly over the entire surface. In fact, once the transformation has occurred, the fine structure associated with the hydride, Fig. 29, could preferentially initiate cathode spots which in turn would lead to a further uptake of deuterium at these sites. This could account for the rather large area, Fig. 28, occupied by these cathode spot remnants.

The fact that we do not observe hydrides in other metals and alloys which have been tested may be due to both the higher solubilities and/or diffusivities of deuterium in those materials as compared to the stainless steels.

4.2 The Effect of Minor Differences in Alloy Chemistry

As mentioned, the presence of low work function impurities is thought to increase the local density of electron emission. Mo has a slightly lower work function than some of the other constituents of 316 stainless and Zr has a slightly higher work function than Nb. The presence of Mo may therefore enhance the emission of electrons in 316 thereby providing more sites for cathode spot initiation and perhaps decreasing the extent of unipolar arcing as compared with Mo free 304 stainless. The opposite effect should be observed for Nb and Nb-751. The 316 stainless steel has, in fact, sustained slightly less area coverage, Fig. 15, and has a slightly higher spot density than 304, Fig. 21. Although the area coverage of Zr free Nb is less than the coverage for Zr containing Nb-751, Fig. 15, the density of remnants in these materials is comparable except after about 600 shots where the density of sites on Nb is greater than that for Nb-751, Fig. 21. Thus, while the results for 304 and 316 appear to agree with the prediction, the results for Nb and Nb-751 do not. The magnitude of the effect, in terms of area coverage, for both the refractory and non-refractory metals and alloys is small, however. It is possible that by increasing the concentration of Mo, or by alloying with elements having work functions, significantly different than the bulk that the area coverage due to cathode spot remnants could be affected significantly.

4.3 The Effect of Systematic Changes in Microstructure

The distinct microstructural conditions produced by the heat treatment of 4130 steel, Figs. 10 and 11, were found to affect the cathode spot remnant distribution, morphology and total area coverage. It appears that microstructural interfaces act as initiating sites for the formation of cathode spots, especially for non-refractory metals and alloys. It is consistent that the remnant area should depend, therefore, on the density of the interfaces intercepting the external surfaces. As shown in Fig. 16 coverage is a maximum for the martensitic condition and a minimum for the austenitized condition with the coverage for the spheroidized condition falling in between. This correlates directly with our estimates of interface density given in EXPERIMENTAL RESULTS.

The distribution of cathode spot remnants is also a function of interface density. For a single plasma exposure, of austenitized 4130, in fact, the only remnants which could be found were associated with pearlite colonies, i.e. the location of the maximum interface density available in this condition. The low exposure data, indicates that the austenitized condition of 4130 has the least tendency to form contiguous cathode spot remnants, i.e. only slight increases in the mean area and density of cathode spot remnants with exposure, as compared to the higher interface density conditions, Figs. 20 and 23. Even after 100 plasma exposures, Fig. 34a, the observed cathode spot remnants were found to be largely restricted to the pearlite colonies. The contribution to the total area coverage of austenitized 4130, by remnants occurring only within ferrite grains and only within pearlite

colonies is shown in Fig. 39 as a function of plasma exposure.

As expected, some cathode spot remnants are indeed observed wholly within ferrite grains but their contribution to the area coverage is always less than 10 percent of the total. The contribution of remnants from 100 to 1000 plasma exposures, associated only with pearlite colonies is, though, at least 50% of the total area coverage.

In terms of morphology the large, fern-like cathode spot remnants observed for the spheroidized, Fig. 35b) and martensitic, Fig. 35, conditions, even after just 100 (10^{18} ions $\cdot$ cm $^{-2}$) plasma exposures is not observed for the austenitized condition until almost 1000 plasma exposures (10^{19} ions $\cdot$ cm $^{-2}$). It is reasonable to expect larger features to occur, at earlier exposure levels, on the microstructural conditions with the higher density of sites. Also these features should sustain rapid apparent growth, i.e. the formation of contiguous cathode spots, due to the high density of initiation sites which are available. As shown in Figs. 20 and 23, the mean area and density of cathode spot remnants for the higher density martensitic and spheroidized conditions increase rapidly as compared to the slight increase in mean area and density observed for the austenitized condition over the first five plasma exposures. As the number of plasma exposures or fluence increases, the austenitized condition exhibits a sustained increase in mean area and density of cathode spot remnants, Figs. 19 and 22, i.e. the number of available sites has yet to be exhausted. The spheroidized condition, on the other hand, shows a comparatively rapid increase in mean cathode spot remnant area,

Fig. 39. Percent area coverage due to cathode spot remnants as a function of exposure for the austenitized condition of 4130. The contribution of remnants observed completely within ferrite grains and completely within pearlite colonies is shown. Note that the ferrite contribution is less than 10 percent of the total.

Fig. 19, but the remnant density after about 100 (10^{18} ions $\cdot$ cm $^{-2}$) exposures, Fig. 22, is a decreasing function of exposure. This apparent decrease is the result of the extensive coverage of the spheroidized condition by overlapping remnants. The coverage for the martensitic condition was so extensive that even at 100 plasma exposures few individual remnants could be resolved. It should be mentioned that we are unable to determine whether or not cathode spots continue to form on specimens like the martensitic condition of 4130 once apparent saturation had been achieved.

It is clear that unipolar arcing can be very dependent on microstructural interfaces. It is not clear however, whether the effect is a function only of the geometry of the boundary, i.e. the step associated with an etched boundary, or whether it is a function only of composition, i.e. due to the segregation of atomic species to boundary sites or whether it is due to a combination of both effects.

5. CONCLUDING REMARKS

In this thesis we have examined the effect of metallurgical variables such as chemistry and structure on unipolar arc damage. It was determined that small amounts of impurities having work functions which differ only slightly from the work function of the major constituents of the alloy resulted in only slight differences in unipolar arc damage. The effect, on unipolar arc damage, of systematic changes in the concentration of an alloying element having, say, a significantly lower work function than the matrix constituents, should be investigated.

In terms of metallurgical structure the results for the non refractory alloys indicate that unipolar arcing is very sensitive to the density of available microstructural interfaces. Refractory metals and alloys, on the other hand, experience unipolar arcing which appears to be largely independent of microstructure. The influence of structure could be further analyzed by determining the arcing tendencies of an alloy as a function of grain size. Changes in the composition of the grain boundaries due to annealing could be monitored by various techniques including microprobe analysis. Ultimately the effect of compositional variations, produced either by segregation to free surfaces or by environmental contamination, on unipolar arcing should be monitored in the shock tube by insitue surface sensitive techniques.

We have shown that thermionic emission may be limited by the short duty cycle of the shock tube. We do not believe, however, that unrestricted thermionic emission would preclude the formation

of erosive cathode spots on any material surface. This could be verified by heating refractory metal specimens in situ in the shock tube and determining the effect of increasing temperature and therefore increasing thermionic electron current on unipolar arc damage.

Although the study of the high temperatures required for significant thermionic emission would resolve whether or not erosive cathode spots form on active thermionic emitters, the study of lower temperatures is also of interest.

The first wall of a fusion reactor is expected to operate at a temperature of about 500°C. The results reported in this thesis are from specimens with an essentially ambient bulk temperature. Work is currently in progress to determine the effect of temperature on the unipolar arc effects which we have observed. It is expected that arcing will be affected through the temperature induced changes in the microstructure. Some microstructures will anneal out, e.g. martensite will decompose, other structures will precipitate out of solution, e.g. carbides stable at higher temperatures, and still other structures may not be altered to any significant degree, e.g. grain boundaries. Temperature variations will not therefore eliminate the dependence of arcing on structure. The dependence of arcing on composition will be affected indirectly by temperature, through the dissolution and precipitation of structural constituents.

The fact that we do not directly measure the erosive products produced by unipolar arcing is a major limitation. Presently, attempts are being made to determine the erosion from non-metallic materials like

graphite, directly using a relatively simple residual gas technique. The direct analysis of the erosion from metallic specimens is more difficult and would require in situ surface sensitive techniques in the plasma shock tube.

An estimate of the erosion resulting from unipolar arcing on the metals and alloys tested can be made, however. The mass loss due to erosive cathode spots has been determined for copper⁴⁰ and is $10^{-4} \text{ g} \cdot \text{coulomb}^{-1}$ or $10^{18} \text{ atoms} \cdot \text{coulomb}^{-1}$. Since these spots are characterized by a current density of $10^6 \text{ amps} \cdot \text{cm}^{-2}$, the mass loss rate, or erosion flux, is about $10^{24} \text{ atoms} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$. The mass loss for cathode spots on stainless steel however, has not been determined. For the purpose of this estimate we assume that it is equal to that for copper. Given our results for the area coverage of stainless steels, about 8 percent coverage after 1000 plasma exposures it can be shown that an impurity concentration of about 10^{14} would result due to unipolar arcing alone during a tokamak discharge.¹⁵ The impurity flux due to arcing is, therefore, on the order of the plasma density itself and is greatly in excess of the concentration of ferrous impurities (about 10^{11} cm^{-3})⁶⁶ estimated to prevent the ignition condition in fusion power reactors.

Certainly the selection of materials for fusion power reactor applications cannot be based merely on the arcing tendency of the material. In addition to large impurity fluxes, which result due to unipolar arcing, the stainless steels, for example, offer an additional

apparent drawback with respect to fusion alloy development. These alloys also sustain degradations of mechanical properties including the appearance of cracks as discussed above, and the loss of tensile ductility reported elsewhere.⁵⁷ The loss of ductility appears to be due to deuterium environment embrittlement. Such degradations in mechanical properties would depend strongly on the temperature of the structure, the time available for diffusion and the availability of hydrogen isotopes.

Other factors which must be considered for, say, a first wall structure include material availability, fabricability, neutron induced after heat, and compatibility with the reactor coolant. Such multi-faceted requirements suggest that the first wall may consist of a coated or a composite material. Irrespective of the actual design of the first wall and given the apparent potency of unipolar arcing as an impurity generating mechanism, the occurrence of unipolar arcs in magnetically confined fusion power reactors clearly must be precluded. Failing that, techniques able to deal with the impurity fluxes produced by unipolar arcing and by other erosive plasma-materials effects, must be devised.

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APPENDIX I:
METALLOGRAPHIC PREPARATION PROCEDURES

<u>Material</u>	<u>Fine Grinding</u>	<u>Polishing</u>	<u>Etching</u>
AISI 304 AISI 316 & Nimonic* PE-16	Mechanical: 400, 600 grit (25,15 μm) Silicon Carbide Abrasive	Mechanical: 0.3 & 0.05 μm Alumina Abrasive	Electrolytic: Note A
AISI 4130	"	Mechanical: 6 & 1 μm Diamond Abrasive	Chemical: saturated picral, by immersion
W A/C	"	Electrolytic: Note B	Electrolytic: Note B
W P/M	"	Electrolytic: Note B	Chemical: Modified Murakami's Reagent: Note C
Nb, Nb-751	"	Chemical/ Mechanical Note D ⁸⁴	Chemical: Note D

* Nimonic is a registered trademark at the H. Wiggins Co. Ltd., Hereford, U.K., a subsidiary of the International Nickel Co., USA.

Note A: Electrolytic: 10% aqueous oxalic acid solution using a stainless steel cathode and passing about 650 mA $\cdot$ cm⁻². Final etch for t < 5 sec.

Note B: Electrolytic: fresh solution of 7 parts methanol, 1 part H₂SO₄, using a stainless steel cathode. Arc cast W was polished at 10 V and etched at 5 V. Powder metallurgical W was polished at 20 V.

Note C: Modified Murakami's Reagent; 15g K₃Fe(CN)₆, 2g NaOH, 100 ml H₂O.

Note D: After fine grinding, specimens are subjected to an intermediate etch: 16 parts lactic acid, 6 parts Hf, 4 parts HNO₃; rough polishing with an alumina slurry: 10 ml 20% chromic acid, 8-10g 0.3 μm alumina and 150 ml H₂O, on a silk cloth over Metcloth** lap at 1150 rpm; intermediate

** Metcloth and microcloth are registered trademarks of the A. Buehler Co Inc., Evanston, IL, USA.

APPENDIX I:

(Continued)

Note D: (continued)

etch again; final polish with alumina paste: 10g $K_3Fe(CN)_6$, 10g NaOH, 100 ml H_2O , 2g 0.05 μm alumina, on Microcloth** at 550 rpm; chemical polish using a solution of 10 ml lactic acid, 10 ml HNO_3 , 5 drops HF; and a final chemical etch using a fresh solution of 7.5 parts lactic acid, 7.5 parts H_2O_2 (30%), 5 parts HF, 5 parts HNO_3 .

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