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Annual Technical Progress Report - I

**"A Study of Grain Boundary Segregation
Using the Auger Electron Emission Technique"**

by

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Annual Technical Progress Report - I**"A Study of Grain Boundary Segregation
Using the Auger Electron Emission Technique"**

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D.F. Stein and P.V. Ramasubramanian**I. Introduction**

The major emphasis during the first eight months of the contract has been on the design, assembly, and check-out of the Auger electron emission system and its components. The system is now operable and producing spectra. Figure 1 shows a photograph of the entire system.

II. Design of System

The high vacuum system was custom made by Varian Associates-Vacuum Division. The system has a large internal volume (Figure 2) sufficient to enable impact fractures of specimens and sputtering of samples in addition to the Auger Electron Emission analysis of fracture surfaces. The fractures will be produced by impacting the specimen with a hammer at the end of a rotary feedthrough. The specimen may be cooled by resting the specimen holder on a copper block cooled by liquid nitrogen. Preliminary tests of the apparatus indicates that temperature as low as 100°K are obtainable using this apparatus. The general layout of specimen holder and manipulator, the cooling blocks, the impacting rotary feedthrough, the sputtering system, and the electron gun and grid system for the Auger electron emission analysis are shown in Figure 3 and the photograph in

Figure 4.

Rough pumping is accomplished by two standard Varian Vac Sorb pumps to eliminate carbon contamination of the system. High vacuum is produced using a standard Varian titanium sublimation pump and a Varian 110 liter/sec noble gas ion pump. The entire vacuum system is bakable to 250°C .

Figure ~~4~~⁵ is a schematic of the Auger electron emission analysis electronics designed by Roland Weber of the Electrical Engineering Department, University of Minnesota. The function of each component and operating procedure is briefly described below:

Grid System:

Consists of a

- (1) a filament for electron supply
- (2) 3 electrode plates for focussing and accelerating the electrons from the filament towards the target

V_1 — draws the electrons away from the filament. V_1 varies from 0-22V. The amount of emission can be controlled by varying V_1 .

V_2 — focusses the electrons towards the target.

V_3 — controls the energy of the electrons leaving the electrode towards the target. Electrons drift at uniform velocity from this electrode and hit the target.

- (3) 4 grids G_1 , G_2 , G_3 and G_4 .

The specimen and G_1 and G_4 are at ground potential. A voltage from 0 to 1000V with a superimposed small constant frequency modulation is swept over the grids G_2 and G_3 .

(Fig. 1)

(Fig. 2)

(Fig. 3)

(Fig. 4)

BLOCK DIAGRAM
(Fig. 5)

Only those secondary electrons which can overcome the negative potential barrier at G_2 can pass through G_2 . The electrons, then are accelerated towards G_4 .

- (4) Collector: This collects all the electrons coming through G_4 . To effect efficient collection, the collector is kept at a positive potential varying from 45 to 495V.

Filament Supply: Harrison D.C. Supply is used for supplying power to the filament for emission. Modifications have been made in the supply so that it can float at a voltage of 2000. The filament draws a current of 0-6A at a voltage that can be varied from 0-6V.

Gun Supply: This keeps the electrodes V_1 , V_2 and V_3 at appropriate potentials. This supply is made up of 7 Eveready batteries 300 V each and one 22 1/2 V battery. V_3 is kept at a potential of 2000V and is grounded. V_2 is at a negative potential of 300-500V with respect to the ground. V_1 and the filament are at a negative potential 2000V with respect to the ground. V_1 controls the emission from the filament through a positive voltage variation from 0-22V with respect to the filament.

Sweep Supply: Keithly high voltage supply is used for continuous sweep of voltage from 0-1000V on the grids G_2 and G_3 .

Programming Control: This enables the achievement a continuous sweep at the desired rates by means of a d.c. motor coupled to the sweep supply. There is also provision for continuous manual sweep.

Oscillator: This superimposes a small modulation at a desired frequency and amplitude over the sweep voltage from the sweep supply. The signal from the oscillator is attenuated to a 10:1 ratio and sent to the Lock-in-amplifier reference.

Collector Bias: This consists of 90V batteries in series and applies a positive bias voltage on the collector to ensure the collection of most of the secondary electrons coming through the grids G_2 and G_3 . The signal from the grid system is passed on to the lock-in amplifier through the collector bias.

Lock-in Amplifier: This gets the reference signal from the oscillator. The signal coming from the collector through the collector bias is amplified and fed into the Houston recorder. Both fundamental and 2nd harmonics can be amplified and fed to the recorder.

Houston-Recorder: This receives its inputs from the sweep supply and lock-in amplifier. X-axis input is from sweep supply through the programming control so that we have the x-axis in volts (electron volts). The amplified output of lock-in amplifier is the input for the y-axis. The unit for the y-axis is arbitrary.

III. Preparations of specimens

Johnson-Matthey spectrographically pure iron is used as the starting material for the iron base alloys. To study the effect of various oxygen and carbon concentrations the following techniques will be used. To produce moderately low carbon and oxygen content we are zone melting the material for 3 passes. This should reduce the carbon and oxygen from the 300 and 100 ppm range (the usual carbon and oxygen content of Johnson and Matthey iron) to near 10 ppm of each or lower.⁽¹⁾ By using the technique of wet hydrogen treatment before zone melting one should be able to maintain the high oxygen while reducing the carbon to a few parts per million. To introduce carbon a simple mixture of a hydrocarbon with hydrogen will be used. These combinations of treatment should enable us to produce a large variation

in carbon and oxygen contents in the specimen. The zone melting is proceeding and the furnace equipment, tubes, and atmosphere controls are in the process of assembly.

The material to be used to study phosphorous embrittlement of iron will be produced by drilling a zone refined iron rod through the center and filling the cavity with powdered phosphorous and re zone melting.

IV. Conclusion

The system to induce fractures in vacuum at low temperatures, to sputter the surfaces produced, and do Auger Electron Emmission analysis of the fractured surfaces or sputtered surfaces has been designed and is now operating. The initial studies will be on a group of several pure metals so we can calibrate the unit in respect to the spectra of the various elements and then we will proceed with the analysis of grain boundary fractures of iron base alloys.

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