APPENDIX A

THE ATOM PROBE FIELD-ION MICROSCOPE

A.1 General Introduction

The Atom Probe Field-Ion Microscope (APFIM, reviews e.g. Mueller 1970a, 1974; Panitz 1975; Wagner 1980, applications e.g. Ralph and Watts 1977; Waugh and Southon 1977; Brenner 1978; Miller, Beaven and Smith 1979; Brenner and Miller 1980; general bibliography Thurstans and Walls 1980) combines a field-ion microscope (FIM, Mueller 1951, reviews e.g. Mueller and Tsong 1969; 1973 applications e.g. Hochman, Mueller and Ralph 1969; Mueller 1970b; Ralph 1970; Hasiguti et al. 1977) with a time-of-flight mass spectrometer (see figure A.1). This permits microanalysis on an atomic scale of selected microstructural features.

The specimen is a sharply pointed tip of ~20nm diameter held at a high potential of +(5-25)kV. The extremely high electric fields which result at protruding atoms are sufficient to ionize imaging gas. An electron tunnels into the tip and a positive gas ion is released. This ion travels radially away from the specimen towards a detector (phosphor screen or channel-plate) where the sum total of events gives an approximately stereographic projection of surface crystal structure. Resolution is of the order of 0.2-0.4nm.

Alternatively, the surface atoms themselves may be ionized at higher applied potentials. In APFIM a pulsed voltage is employed to release ions at a given time and the time of flight over a selected

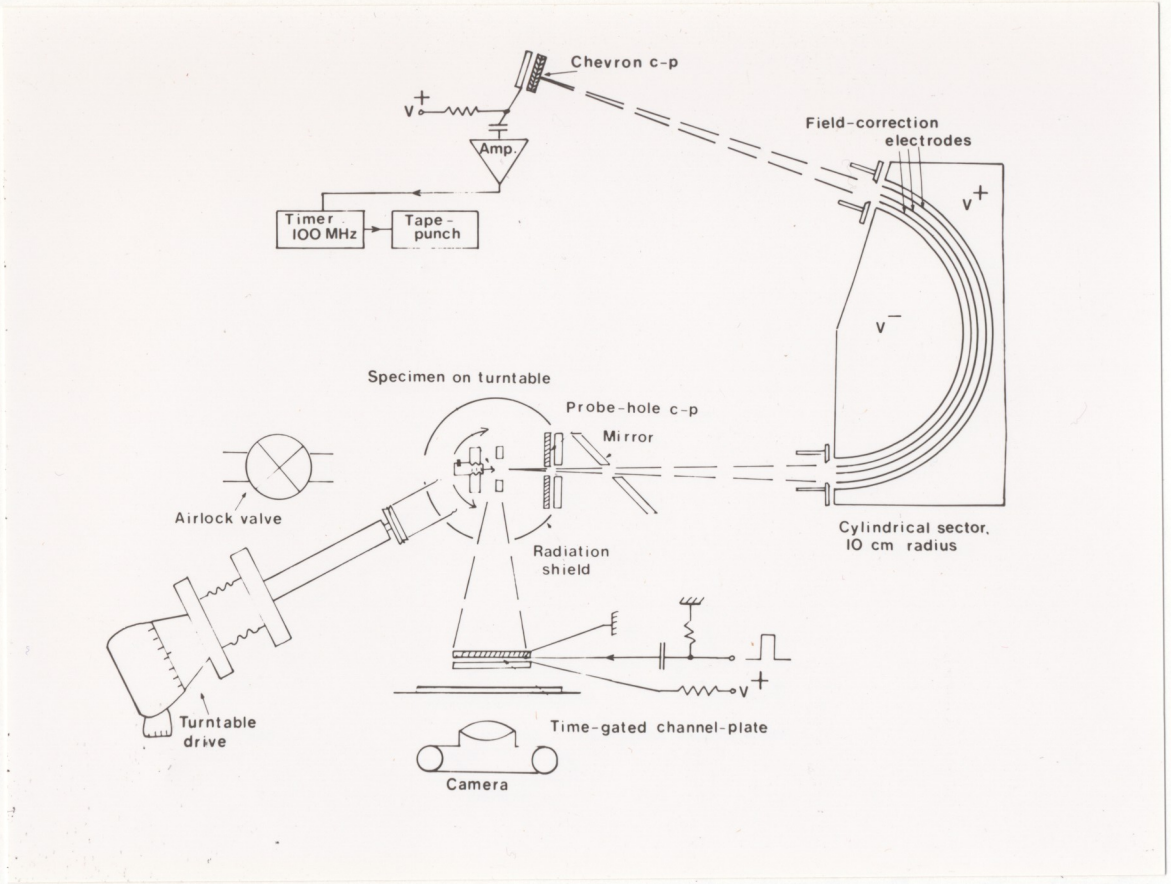


Figure A.1 Schematic diagram of the combined energy-compensated and imaging atom-probe used to obtain some of the data in the present study (courtesy of Dr. A. R. Waugh).

drift distance is measured. The mass-to-charge ratio is then obtained from the time of flight using the equation

$$\frac{m}{ne} = 2 (V_{DC} + V_P) t^2/L^2 \qquad \text{....EA.1}$$

In the original quantitative atom probe (QAP, Mueller, Panitz and McLane 1968) a straight drift tube was employed. Subsequently an energy-compensated configuration (Poschenrieder 1972) and magnetic sector atom probes (Mueller and Sakurai 1974) were developed. Mass resolution in these latter instruments is in the range 0.50-0.05a.m.u.

For QAP measurements a small area of the specimen is selected for analysis using a probe hole aperture in the imaging assembly (figure A.1). As the specimen is evaporated (ionized) this aperture generally samples 25-200 ions from each successive atomic plane. Thus a profile of compostion against probing distance may be compiled (see Chapter 3 of this dissertation).

Alternatively a two-dimensional map of elemental distributions in the plane perpendicular to the probing axis may be produced (IAP). In this case the channel-plate normally used to intensify the gaseous field-ion image is employed as detector for evaporated metal ions (Panitz 1973, 1975). The electron multiplier is gated on for a short period after a delay corresponding to the drift time of the required species. Events from several successive planes are recorded photographically. The field of view searched by IAP is approximately

30nm, with resolution of ~1.0nm.

A.2 Present configurations

The majority of QAP traces were obtained using a straight flight tube instrument originally designed and built by A. J. Watts (Watts 1975; also Watts and Ralph 1978). The major modifications of this APFIM were the addition of a 250Hz pulser (Waugh 1980) and updating of the computer-aided data collection system. These changes raised the rate of data collection from ~4000 to ~10000-12000 ions per hour.

In operation, flight time data were collected automatically from a two-channel Schottky logic timer (Watts 1975) and stored on floppy disc by a CE 8000 microprocessor. The results were then transferred to magnetic tape using a graphics-designated PDP 11/45 of the University of Cambridge Computing Service (UCS). Subsequent processing of flight times was accomplished using an IBM 370/165. The help of R. Laborde (UCS) and D. I. Singer (UCS) in construction and operation of the PDP 11/45 link is gratefully acknowledged.

Subsidiary QAP analyses and all IAP determinations were performed on a combined QAP(energy compensated)/IAP designed and built by A. R. Waugh (Waugh 1978; Waugh and Southon 1979).

Table B.1 Melt Sources

System	Source	Materials	Method	Additional data
Ni-Al	a) International Nickel Co. Ltd. (Melt A).	High purity elements	Supplied as forged bar	
	b) Drs. J.V. Bee J.V. Wood MMSC	Inco. melt A	Melt-spinning	APFIM wire only
Ni-Cr-Al	Mr. Leader MMSC	Inco. melt A + high purity chromium and aluminium	Multi-pass arc	
Ni-Al-Ti (2 alloys)	Mr. J. Leader MMSC	Inco.melt A. + high purity nickel bar and sponge titanium	Multi-pass arc	
PE16 matrix model (supplied by Dr. M.P. Shaw, MMSC)	Mr. J. Leader MMSC	High purity elements	Multi-pass arc.	Rolling by U.K.A.E.A. Harwell

* MMSC - Department of Metallurgy and Materials Science, University of Cambridge.

Table B.2 Heat Treaments

Composition (wet analysis)/at%	Solution Treatment Temp/°C	Solution Treatment Time/hrs.	Ageing Treatment Temp/°C	Ageing Treatment Time/hrs.
Ni -14at% Al(Inco.)	1250±10	48	625±8	0-1000
Ni -14at% Al melt-spun	As-received only		-	-
Ni - 16.7at%Cr-14.5at%Al Ni -20.0at%Cr -14.0at%Al	1252±10	2	620±10	0-2
	1350±15	2	625±10	0-5
Ni -9.1at%Al -4.4at%Ti	1250±10	2	-	-
	1050±10	2	-	-
Ni -8.7at%Al -2.5at%Ti	1250±10	2	625±10	0-5
PE16 model see Table 6.1	1050±10	2	900±10 followed by 750±10	1 0-8

APPENDIX B

SPECIMEN PREPARATION AND EXPERIMENTAL CONDITIONS

B.1 Specimen Preparation

B.1.1 Melts

Details of the required alloy compositions and melt preparations are given in Table B.1.

Solution and ageing treatments (Table B.2) were performed upon field-ion wires (250μm * 250μm * 10mm) and TEM discs (3mm diameter * 250μm thick) slit from the melts after initial homogenization. The use of such small specimens was considered to be permissible for these alloys because decomposition products occur on a fine scale of 5-20nm.

With the exception of very short ageing sequences (2-20 minutes) for the nickel-chromium-aluminium alloy, all heat treatments were performed upon specimens sealed in silica capsules with 80 mm pressure dry argon. In the case of the nickel-chromium-aluminium it was considered that the time required for the capsule to attain temperature would result in significant reduction of true ageing time. These specimens were therefore wrapped in titanium foil for oxygen gettering and heat treated in an argon stream. Rapid quenching was accomplished into iced brine.

The results of wet chemical analyses of the melts are also given

Tables B Polishing Conditions

Table B.3.I. APFIM specimens (room temperature)

Alloy	Solution	Voltage/V	Solution	Voltage/V
Ni -14at%Al	15% perchloric acid/acetic acid	30 D.C.	2% perchloric acid/2-butoxyethanol	8 D.C.
Ni -16.7at%Cr -14.5at%Al	"	"	"	10 D.C.
Ni -9.1at%Al -4.4at%Ti Ni -8.7at%Al -2.5at%Ti	"	"	"	10 D.C.
PE16 model	"	"	"	8 D.C.

Tables B Polishing Conditions

Table B.3.II. TEM specimens

Alloy	Solution	Voltage/V	Temp/°C
Ni -14at%Al	2% perchloric acid/ 2-butoxyethanol	75 D.C.	-5
Ni -16.7at%Cr -14.5at%Al	4% perchloric acid/ 2-butoxyethanol	115 D.C.	-10
Ni -9.1at%Al -4.4at%Ti) Ni -8.7at%Al -2.5at%Ti)	2-5% perchloric acid/ 2-butoxyethanol	95-115 D.C.	-10

in Table B.2.

B.1.2 Polishing conditions

APFIM specimens were lightly abraded to remove surface oxide film before electropolishing. TEM specimens were ground to a thickness of 100 μm. The polishing conditions employed are detailed in Table B.3.

Specimens of the binary nickel-aluminium alloy were also outgassed at 200°C for 1 minute subsequent to electropolishing.

B.2 Experimental conditions

B.2.1 Atom Probe Microscopy

The atom probes employed in the study are described in detail in Appendix A. QAP and IAP analyses were performed with background vacua of ~10^{-9}Torr and at 78K. Output pulse:standing voltage ratios in the range 16-20% were employed.

B.2.2 Transmission Electron Microscopy

TEM studies were made using Philips EM300(100kV) and EM301(120kV) instruments.

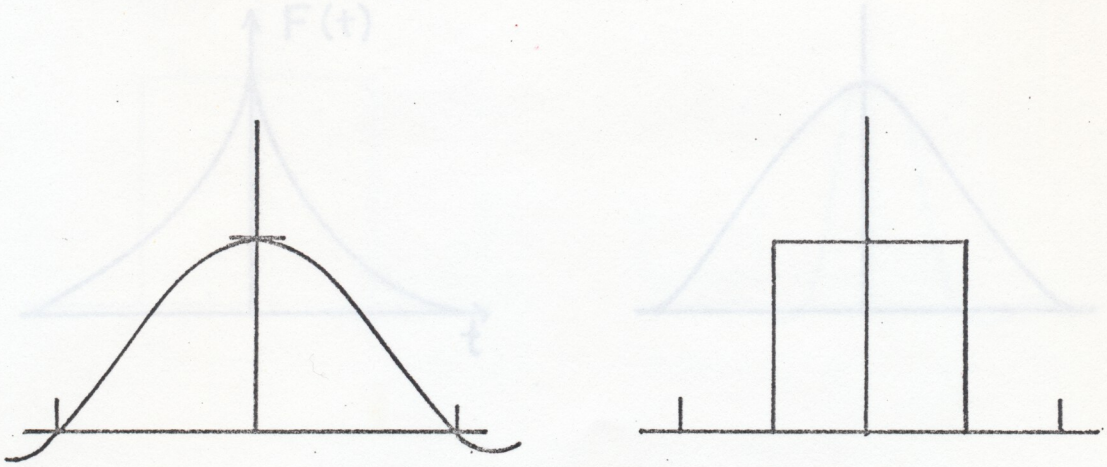
B.2.3 X-ray Powder Diffraction

X-ray powder photographs were obtained in the Debye-Scherrer setting. CuK_α radiation was employed throughout. Exposure times were: nickel-aluminium 3 hours at 100W; nickel-chromium-aluminum 2.5 hours at 150W; PE16 matrix model 2.5-4.0 hours at 100W.

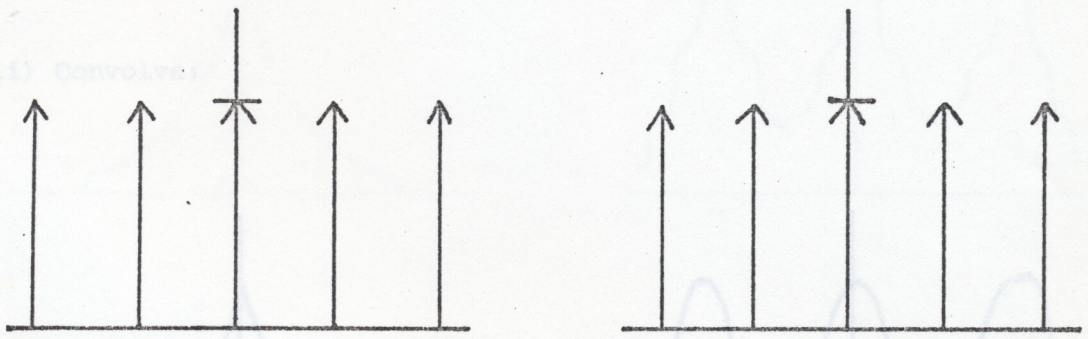
APPENDIX C

C.1 Special functions and their transforms.

i) Rectangular sampling function $\Pi(t)$:

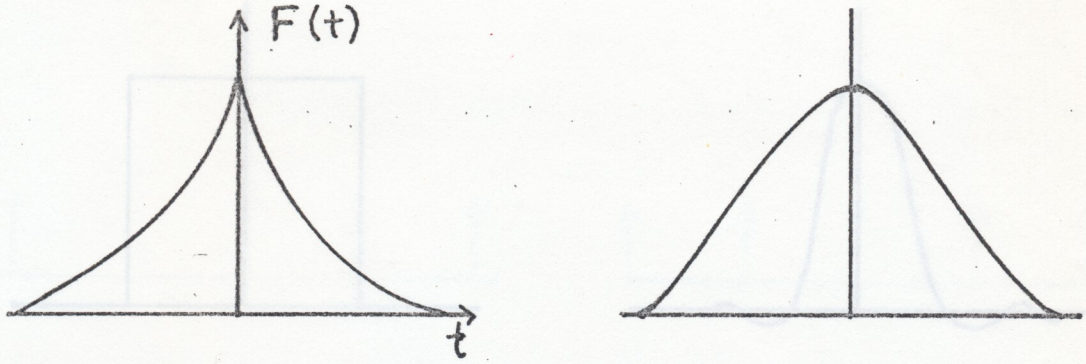


ii) Infinite impulse train III:

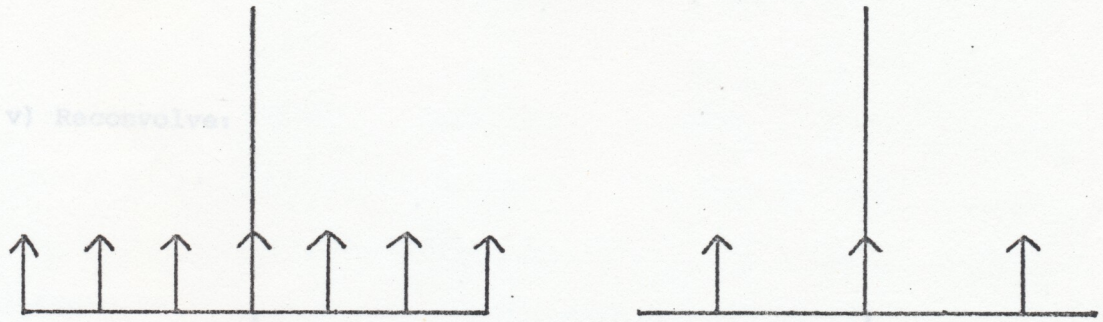


C.2 Stages in Fourier transforming experimental data.

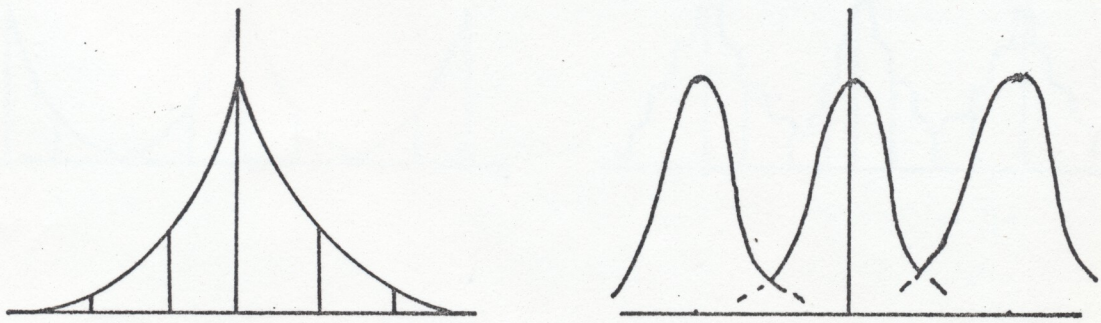
i) General function: (after Brigham 1974)



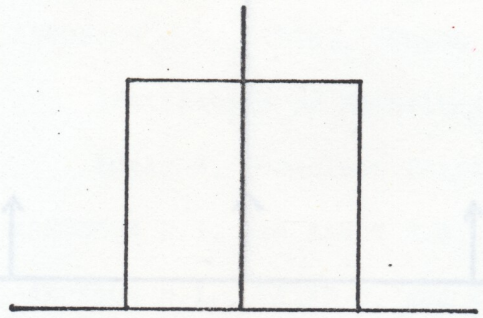
ii) Sample:

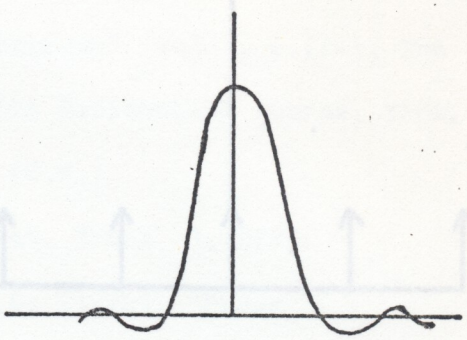


iii) Convolve:

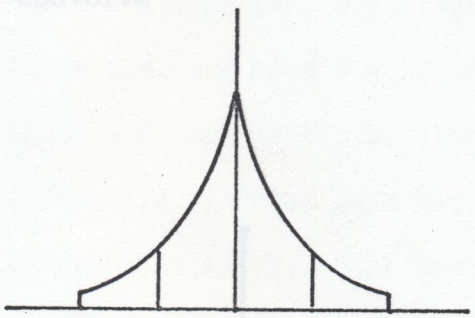


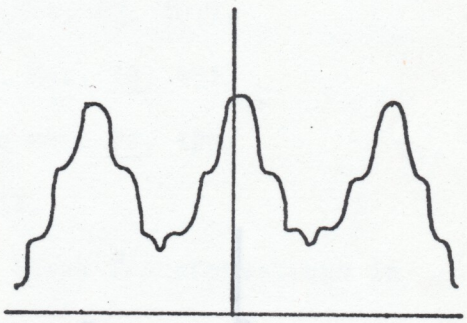
iv) Truncate:



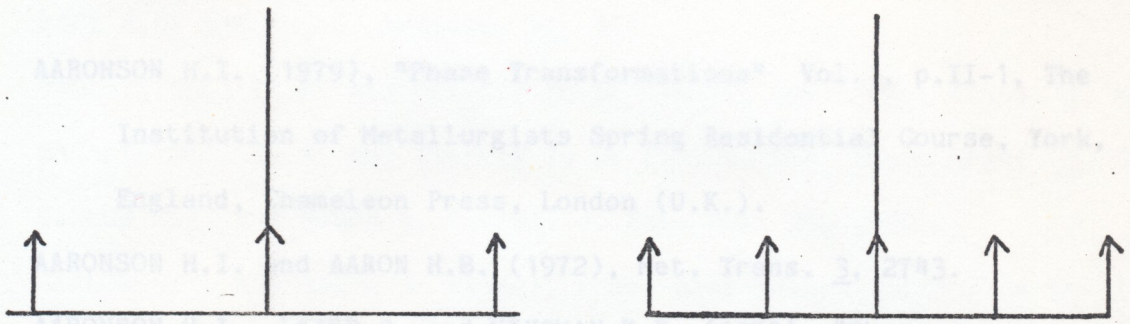


v) Reconvolve:

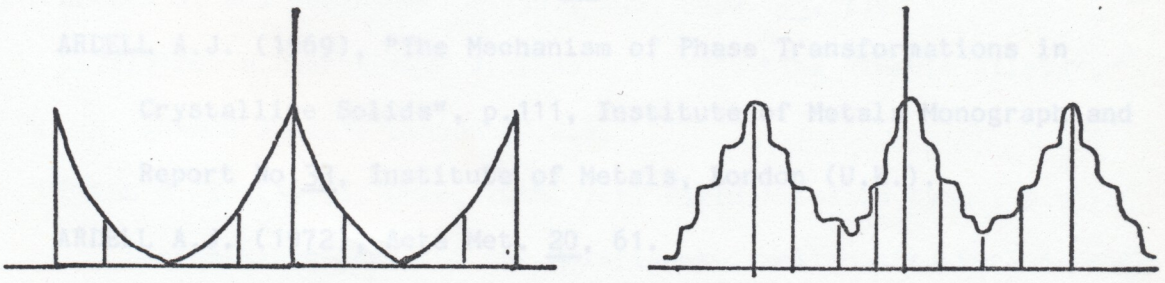




vi) Sample frequency:



vii) Convolve



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