

X-ray Fluorescence Spectrometer for Thin Film Evaluation





# **Flexible, Precision Hardware Features**

### Features of WDX System

Feature comparison between AZX 400 and ED-XRF

	AZX 400 (WD-XRF)	Ordinary ED-XRF		
Analysis target	Be to U	Mg to U		
Resolution (FWHM)*	26 eV	>150 eV		
Analyzable thickness	0.01 nm or greater	Several-10 nm and more		
* In the case of Mn-K				

WDX with high resolution is suitable for Al ultrathin films by separating the Al and Si peaks.



#### Various Sample Adapters

Various sample types can be analyzed simply by changing the adapter. The multi-adapter allows coupons or target materials (see the adapter at top left or bottom left in the photo).



## **CCD** Camera

The optional CCD camera allows pinpoint measurements of defects and/or patterns and enables analyses at 0.1 mm pitch.



2 cm

5 mm

## Wafer Loader

A 300 mm FOUP or open cassette down to 100 mm are available for the wafer loader. This system enables automatic analyses and reduces users' handling time dramatically.



## Diffraction Elimination

In analyses of single-crystal wafers, diffraction peaks often affect the target spectrum and cause errors. AZX 400 eliminates undesirable diffraction peaks using filters for accurate results. Six types of filters enable the analyses of all elements.

Diffraction peaks overlapping Ta-L $\beta$ 1







Without filter

With filter

# **Useful Software Features**

## **Quantitative Application Setting Flow for Thin Film**

The software supports easy calibration curve setup, even for first-time users.



#### Standardless Analysis Using Sensitivity Library

If standard samples are hardly available, standardless analysis can be performed by using the optional SQX software. This function is available both for the thickness analyses for thin films and material composition analyses for bulk samples.

#### AZX 400 standardless analysis accuracy

Analysis element * 1	AZX 400 standardless analysis value (nm)	Standard value of thin film standard sample *2 (nm)
Cu	52.90	54.36
Ni	47.73	49.55
Pt	20.75	21.31
Au	23.80	23.99

The error rate is about 5% for single-layer analyses of the thickness. It is efficient to analyze estimated value especially when a standard sample is not available. If necessary, the correction function can be used to manage analysis values.

- \* 1 Analysis element in single-layer film on polymer
- \*2 Thin film standard samples for XRF available from MICROMATTER

The amount of adhering material is converted into a film thickness value using the density values stored in AZX software. Errors in standard values are within  $\pm$  5%.

SQX analysis of stainless steel (JSS651-11)



					Unit: mass%
Component	Quantitative value	Standard value	Component	Quantitative value	Standard value
Si	0.42	0.42	Co	0.18	0.17
Р	0.031	0.035	Ni	10.45	10.11
Cr	19.11	18.47	Cu	0.40	0.39
Mn	1.91	1.70	Мо	0.18	0.16

# A Flexible Technique for a Wide Variety of Applic

### MgO Ultra-thin Film Analysis

MgO film thickness analysis can be performed accurately in units of 0.1 nm. Since the L.L.D. will be smaller than 0.01 nm, it is possible to analyze ultra-thin films of less than 1 nm.

Theoretical analysis value of MgO ultra-thin film (0.5 nm)

Film thickness (nm)	0.5
Range (nm)	0.014
Std. Dev. (nm)	0.007
R.S.D. (%)	1.43

 Figures are calculated values based on theoretical X-ray intensity. (Measurement time: 60 sec)

High-sensitivity analysis enables accurate measurement of differences as small as 0.1 nm.





\* Measurements for thicknesses ranging from 1 to 2.5 nm were obtained at Rigaku Lab. The value indicated for 0.5 nm is a simulated intensity.

\* Ta is used for cap layer and/or barrier layer.

#### Thickness/Composition Simultaneous Analysis of CoFeB Magnetic Layer

Widely used in magnetic thin films for universal memory devices and magnetic heads, B can be difficult to analyze. AZX 400 with Fundamental Parameters (FP) method makes it possible to simultaneously analyze both film thickness and material composition.





Results of 10-times repeatability analysis of CoFeB layer

	Film	Film Material compo			
	thickness	Co	Fe	В	
Target value	5	30	50	20	
Unit	nm	at%	at%	at%	
Average	5.01	29.99	49.94	20.07	
Range	0.07	0.49	0.65	0.31	
Std. Dev.	0.020	0.14	0.18	0.09	
R.S.D.(%)	0.41	0.47	0.37	0.45	

### Multilayer Analysis of STT-MRAM

AZX 400 can analyze up to 10 layers of different components. In the case of STT-MRAM, it analyzes not just a single MgO or CoFeB layer, but the thicknesses of all layers, as shown below.

Example of STT-MRAM Results of 10-times repeatability analysis of STT-MRAM film stack

	Layer	Ru	CoFeB	MgO	PtMn	Та	Cu
	Target thickness (nm)	8	5	1.5	12	20	40
	Measurement time (sec)	20	10	60	30	10	10
er	Average (nm)	7.99	5.01	1.51	12.00	20.05	39.98
r	Range (nm)	0.09	0.06	0.02	0.15	0.25	0.33
er	Std. Dev. (nm)	0.030	0.025	0.008	0.057	0.098	0.11
er	R.S.D.(%)	0.37	0.49	0.54	0.48	0.49	0.28
r							

 $\ast$  Two or more layers of the same type are analyzed as one layer.

\* Allows CoFeB film thickness analysis in the case of multi-layered film.

layer structure	
Ru layer	
Ta layer	
CoFeB layer	
MgO layer	
CoFeB layer	
Ru layer	
CoFeB layer	
PtMn layer	
Ta layer	
Cu layer	
Ta layer	
Cu laver	

Ta layer

# ations

## 🖉 Au/Ni/Ti/Al-Si (Backside Electrode) Analysis

Since the FP method enables analyses using one calibration curve for each element, analyses can be performed even with a small number of standard samples. (When the Au or Ni film is thick, measurements of Al may not be possible due to X-ray absorption. In those cases, analyses requires two separated processes: one for AuNiTi and the other for Al.)



χ	Results of	10-times	repeatability	analysis of	Au/Ni/Ti/Al-Si
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Layer	Au	Ni	Ti	Al-Si
Target thickness (nm)	50	700	200	200
Average (nm)	50.06	700.24	198.61	199.90
Range (nm)	0.35	1.92	1.80	3.41
Std. Dev. (nm)	0.12	0.55	0.59	0.95
R.S.D.(%)	0.25	0.08	0.30	0.47

\* Si concentration is set as a fixed value.

\* Measurement with 30 mm diameter

#### Thickness/Composition Simultaneous Analysis of PZT (PbZrTi) Film

AZX 400 can perform simultaneous analysis of the film thickness and material composition of PZT, an important material for MEMS and memory devices. In the case of film stacks of Ir or Pt, spectra overlap in a complex manner. Under these circumstances, high-resolution WDX permits effective analyses.

Pb-Lβ1	Zr-Ka Ti-Ka	Results of 10-tim	nes repeata	bility anal	ysis of PZ	T film
			Film	Mater	ria <mark>l c</mark> ompo	sition
PZT			thickness	PbO	ZrO <sub>2</sub>	TiO <sub>2</sub>
Pt		Target value	3,000	50	25	25
		Unit	nm	at%	at%	at%
Ti		Average	3001.9	50.0	25.0	25.0
SiO <sub>2</sub>		Range	20.1	0.2	0.2	0.2
		Std. Dev.	6.59	0.067	0.066	0.061
Si sub.		R.S.D.(%)	0.22	0.13	0.26	0.24

\* Measurement 10 mm diameter

\* Material compositions are converted to oxidative values.

### Analysis of Ag Concentration in Solder Bumps

A solder bump ranges from 10  $\mu$ m to 100  $\mu$ m in size. Ag-K $\alpha$  and Sn-K $\alpha$  should be used to obtain information from a deeper part. If a standard sample is not available, the FP method enables use of Ag and Sn pure metals as standard samples.



Results of 10-times repeatability analysis of solder bump metal

	Concentration				
Element	Sn	Ag			
Unit	mass%	mass%			
Average	98.34	1.66			
Range	0.02	0.02			
Std. Dev.	0.0070	0.0070			
R.S.D.(%)	0.01	0.42			

\*Measurement with 30 mm diameter

#### **Specifications**

X-ray tube	Rh target, maximum rating: 4 kW	Measurement range	SC: 5°to 118°, F-PC: 13°to 148°	
	High-frequency inverter system		1400°/min (2theta)	
X-ray high voltage generator	Maximum rating: 4 kW, 60 kV-150 mA	Angle reproducibility	Within $\pm 0.0005^{\circ}$	
voltage generator	Stability: $\pm$ 0.005% (with 10% input variability)	Continuous scan	0.1° to 240°/min	
Maximum sample	400 (dia.) x 50 (H) mm (30 kg or less)	Crystal exchanger	Automatic 10-crystal exchanger	
size			Standard : LiF(200)、Ge、PET、RX25	
Analysis element applicable	4Be to 92U	Analyzing crystal	Optional : LiF(220)、RX4、RX9、RX35、 RX40、RX45、RX61、RX75	
Sample rotation	5 rpm	Detector	For heavy elements: SC; for light elements: F-PC; with	
Primary beam filter	Standard: AI (2 types), Cu, Zr		automatic center wire cleaning mechanism	
r milary beam mer	Optional: Ti, Sn	Vacuum exhaust	Manuum numn 1 ant	
	Automatic 7 positions exchanger	system	Vacuum pump, 1 set	
Diaphragm	30, 20, 10, 1, and 0.5 mm (diameter)	Vacuum control	APC (Auto Pressure Control), with 3 levels	
With attenuator (X-ray intensity: 1/10): 30, 20 mm (diameter)		Temperature stabilizer	36.5 ℃± 0.1 ℃	
Primary soller slit	Automatic 3 positions changer mechanism standard and high resolution, ultra coarse (optional)	Data processing	Windows PC, printer Software: film thickness/concentration simultaneous	
Secondary soller slit	For SC and F-PC	system	analysis software, Fundamental Parameter software for	
Goniometer	$\theta$ - 2 $\theta$ independent drive system		thin film analyses	

#### Layout plan and dimensions \*1



\*1 If process modules are installed in a clean room, utility equipment should be in a separated maintenance area.

\*2 Standard model manufactured by Rigaku Corporation

#### Installation requirements \*

Power supply	3-phase 200 VAC, 50A
Ground	Independent grounding with resistance 30 $\Omega$ or less
Cooling water	Temperature: 5 to 30°C Pressure: 0.29 to 0.49 MPa Flow rate:10 L/min Quality: Equivalent to tap water quality Drain: Open drainage Connector (IN/OUT): Joint for 26 (dia.) x 19 (dia.) blade hose Compression fitting (Abe Machinery B121926)
High-purity nitrogen(UPN)	Temperature: 30°C or lower Pressure: 0.52 to 0.7 MPa Flow rate: 200 L/min Connector (IN/OUT): 1/4" Swagelok joint
Adsorption vacuum	Pressure: - 80 kPa Flow rate: 60 L/min Connector (IN/OUT): 1/4" Swagelok joint
Room temperature	15 to 28°C, fluctuation range: within $\pm$ 2°C
Humidity	75%RH or lower
Vibration	Lower than human sensitive level
P-10 gas	Mixed gas of 90% argon and 10% methane Pressure: 0.15 MPa

\*The customer is responsible for connecting utilities to the equipment.



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