APPLICATION NOTE

No. AN41125

Investigation of ceramic materials in 5G Antenna development with ARL EQUINOX 100 XRD and QUANT'X EDXRF

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Introduction

As the demand for wireless data transmission increases year over year, the need for faster speeds and higher volumes continues to rise. The sub 6 GHz range for transmission is already overcrowded, so the mostly unused spectrum above 10 GHz is being examined for the 5th generation wireless data transfer platform (5G). The design of ceramic antenna materials for this specific region of the electromagnetic spectrum is so challenging because temperature stable materials with low dielectric constants (ε'=6-18) and a high Qf (narrow bandwidth) or ultra-high Qf and ε' < 30 are required. There are few classes of materials which could come into consideration, e.g. Eucryptite ($LiAlSiO_4$), Li_2MgSiO_4, Forsterite (Mg_2SiO_4) or Spinel and Perovskite type materials. Also, mixtures and matrix materials as well as dopants are developed as grain boundary effects and vacancies are influencing the properties crucial for antenna usage.

To ensure the quality and performance of the product it is crucial to control chemical and structural composition, not only for mixtures but also for pristine compounds. The easiest and most convenient solution for routine QC/QA processes as well as more sophisticated analyses in research labs is the combination of both energy dispersive X-ray fluorescence spectroscopy (EDXRF) and X-ray diffraction (XRD).

Instrument

The Thermo Scientific™ ARL™ EQUINOX Series represent a portfolio of XRD instruments from simple, easy to use bench-top systems for routine analysis to more advanced floor-standing, research grade systems.

The Thermo Scientific™ ARL™ EQUINOX 100 X-ray Diffractometer employs a custom-designed 50 W (Cu or Mo) or 15 W Co high-brilliance micro-focus tube with mirror optics which does not require an external water chiller. The unit can be transported between laboratories or into the field without requiring any special infrastructure.

The ARL EQUINOX 100 (c.f. Figure 1) provides very fast data collection compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time.

Figure 1: ARL EQUINOX 100 diffraction system

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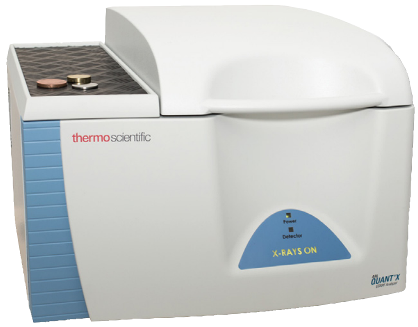
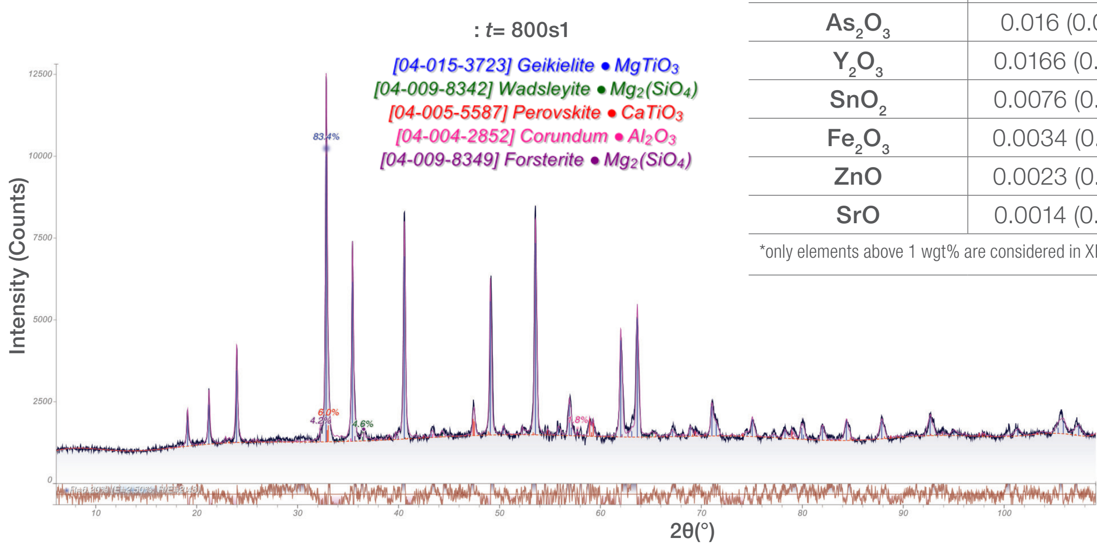


Figure 2:. ARL QUANT'X EDXRF instrument

The Thermo Scientific™ ARL™ QUANT'X Energy-Dispersive XRF Spectrometer uses a highly sensitive silicon drift detector (SDD) to discriminate between the energy of the incoming radiation and therefore is able to measure all elements between Na (Z = 11) and U (Z = 92). It is equipped with a 50 W Rh or Ag tube which can be operated at voltages up to 50 kV. Conversion of spectra into elemental/ oxide concentrations is achieved with the Fundamental Parameters (FP) based UniQuant standard-less package. The rugged and compact design as well as low demand on peripheral support make the ARL™ QUANT'X a perfect solution for industrial environments.

Experimental

A 5G antenna ceramic was measured for 13 min using an ARL EQUINOX 100 with Cu-Kα radiation (1.541874 A). The sample was milled to a fine powder prior to analysis. Data processing and evaluation was performed using MDI JADE 2010 (WPF) equipped with the pdf4+ database.



Results

Measurement in reflection setup followed by phase qualification and quantification (WPF) yield a phase composition consisting of two different polymorphs of Mg_2SiO_4 (Wadsleyite and Forsterite) as well as Geikielite ($MgTiO_3$) and Pervoskite ($CaTiO_3$) accompanied by slight amounts of Corundum ($Al2O_3$) (c.f. Table 1). From the XRD quantification it is possible to calculate the elemental composition and compare it with results from the EDXRF elemental analysis which additionally reveals trace elements (c.f. Table 2).

Table 1: Results of the XRD quantification

Phase	Formula	Content (in wgt %)
Geikielite	$MgTiO_3$	83.4 (1.3)
Perovskite	$CaTiO_3$	6.0 (0.6)
Wadsleyite	Mg_2SiO_4	4.6 (0.7)
Forsterite	Mg_2SiO_4	4.2 (0.6)
Corundum	Al_2O_3	1.8 (0.3)

Table 2: Comparison XRF and XRD

Element*	EDXRF (in wgt %)	XRD (in wgt %)
TiO_2	56.9 (0.48)	59.0
MgO	33.58 (0.24)	33.0
CaO	3.27 (0.09)	2.5
SiO_2	2.58 (0.08)	3.8
Al_2O_3	2.57 (0.08)	1.8
ZrO_2	0.332 (0.017)	-
SO_3	0.242 (0.013)	-
Nb_2O_3	0.215(0.010)	-
NiO	0.179 (0.008)	-
Cl	0.077 (0.004)	-
As_2O_3	0.016 (0.0009)	-
Y_2O_3	0.0166 (0.0009)	-
SnO_2	0.0076 (0.0004)	-
Fe_2O_3	0.0034 (0.0005)	-
ZnO	0.0023 (0.0003)	-
SrO	0.0014 (0.0002)	-

*only elements above 1 wgt% are considered in XRD and above 0.001 wgt% for XRF thermo scientific

Conclusion

Using the ARL EQUINOX 100 it is possible to obtain data suitable for qualitative and quantitative (WPF) phase analysis. The 5G antenna ceramic sample contains phases which are desirable for the usage as antenna in the frequency range above 10 GHz.

EDXRF yields the chemical composition which enables the user to fully control the quality of the product and additionally track concentrations of dopants or pollutants. Results from both XRD and XRF are in good agreement.

Combining XRD investigations with EDXRF is an easy-to-use solution for both industrial and academic research. It allows fast and easy QC/QA procedures, even for untrained operators.

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