APPLICATION NOTE

Determination of the amorphous content in quartz and glass mixtures using ARL EQUINOX 100 X-ray diffractometer

Introduction

In various industrial application the amorphous content of a mixture of compounds significantly influences the properties. In pharmaceutical preparations the crystallinity is crucial for the bio-availability of an agent and therefore it is important to know the amorphous content of such mixtures. The amorphous content of bulk metallic glasses (BMG) is a measure for the physical properties of the compound and therefore for the quality of the product, comparable to cements and slags. One easy-to-use method for the determination of the amorphous content is X-ray diffraction (XRD).

Instrument

The Thermo Scientific™ ARL™ EQUINOX 100 employs a custom-designed Cu (50 W) or Co (15 W) micro-focus X-ray tube with mirror optics. The low power consumed by the unit allows it to be completely tranportable, not requiring an external water chiller. The same unit is capable of being transported between laboratories without the need for special infrastructure.

Figure 1: ARL EQUINOX 100 X-ray diffractometer

The ARL EQUINOX 100 provides very fast data collection times compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time and is therefore well suited for both reflection and transmission measurements (Figure 1).

Experimental

Ball milled (3 min, 20 Hz) mixtures of glass and quartz (wgt% quartz 10,30,50,70) were measured in reflection mode using Cu Kα (1.541874 Å) radiation for 3 minutes with the sample rotating during the analysis. The amorphous content was determined using standard-less deconvolution approaches implemented in MAUD and JADE.

Results

For amorphous contents ≥ 40% the deviation from the weight portion is ±1.5% with both MAUD and JADE, whereas for lower contents the deviation increases (Figure 2). The differences are most likely due to slightly different algorithm used by the programs and the susceptibility to errors of the FWHM determined according to ASTM D5187.

Figure 2: Rietveld fit (JADE) of a 90% glass / 10% quartz sample

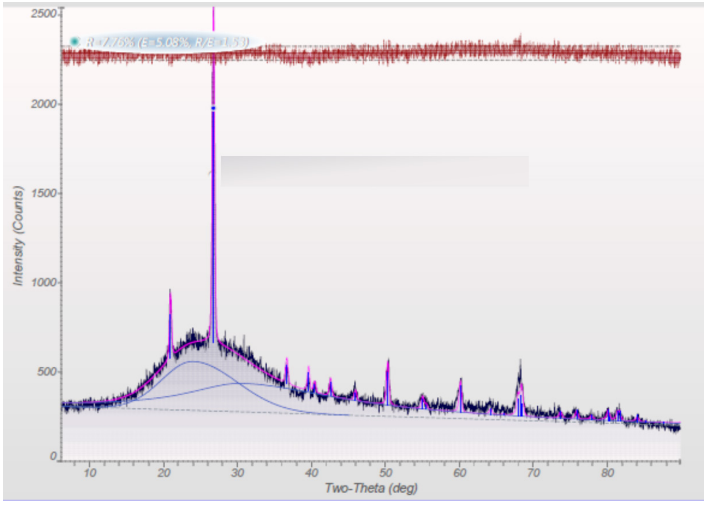


Figure 3: Rietveld fit (MAUD) of a 90% glass / 10% quartz sample

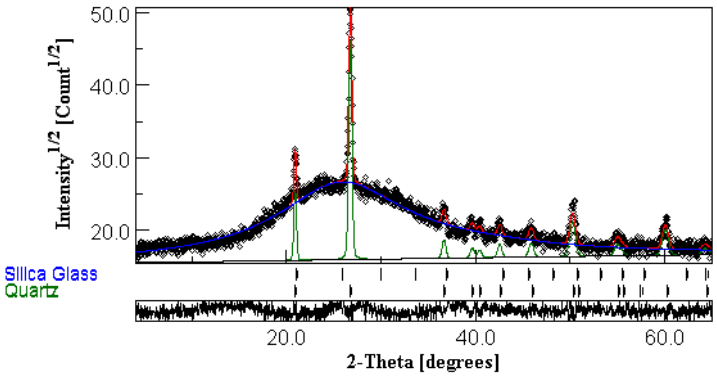
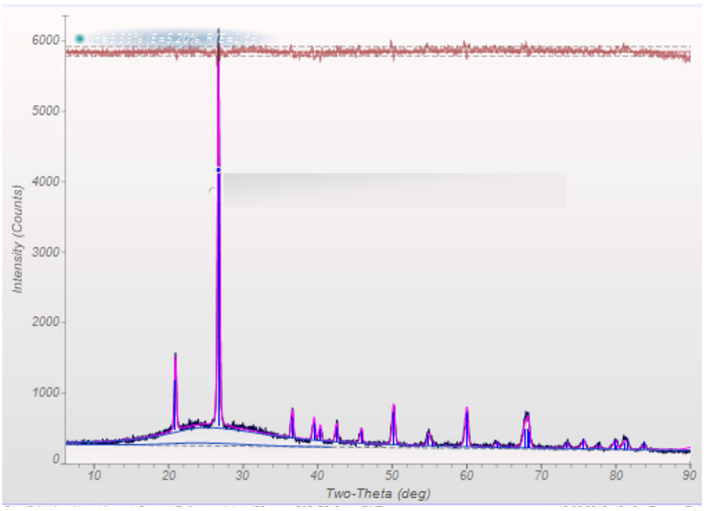


Figure 4: Rietveld fit (JADE) of a 70% glass / 30% quartz sample



Conclusion

The ARL EQUINOX 100 is a suitable instrument to evaluate the amorphous content in glass and quartz mixtures applying a standard-less deconvolution approach using MAUD or JADE with reasonable precision down to ~25 % amorphous content. For lower content a spiking method is recommended.

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