

NEW SAMPLE PREPARATION FOR TXRF ANALYSIS USING RESIST PATTERN LAYER

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Total reflection XRF (TXRF) is well known technique for trace elemental analysis. A small volume droplet of the sample solution is dropped on a flat substrate. After it is dried, the dried residue is analyzed by TXRF instrument. Quantification is performed by standard addition technique. However, the size or shape of the dried residues still influence on the quantitative values. In addition, it was a practical problem to take a long time for drying process. To solve these problems, Prof. Fittschen et.al developed a picoliter solution deposition technique using an ink-jet printer [1,2]. The size of deposits was typically 5 – 20 μm . This sample preparation is useful for TXRF quantification. It is also reported that shading effect is important for TXRF analysis of residues [3]. They confirmed the shading effect directly by using a color x-ray camera under total reflection condition.

We tried to prepare a film type residue. For this purpose, a glass substrate was modified using photoresist pattern. A structure of parallel crosses shape was produced by photo lithography. A 10 μl volume of the standard solution (including Ti, Cr, Ni, and Pb) was dropped on the resist pattern substrate. The dried residue was obtained in a relatively large area of 3-4 mm in diameter. The surface morphology was observed by a laser 3D microscope. The thickness of the residue was thin less than 1 μm . Therefore, a small absorption effect in the residue was expected.

TXRF analysis was performed at a glancing angle of 0.05 degree by using NANOHUNTER™, (Rigaku Co.). An x-ray tube with Mo target was operated at 50 kV and 0.6 mA. A repeatability and RSD values of quantitative results were improved by applying this sample preparation.

References

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