

Relationships between spontaneous breakage and volume expansion of Nickel Sulfide included in tempered sheet glass

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As many examples, the spontaneous breakage of tempered-sheet glass occurs by the crack extension caused by the volume expansion of Nickel Sulfide inclusion which is associated with the phase transformation from alpha to beta. The volume expansion by beta-phase transformation is about 4% based on the previous investigations [1]. It is considered that the Heat Soak Test is one of the most useful solution for the avoidance of spontaneous breakage in the glass market. However, it is difficult to clarify the crystallographic information of the Nickel Sulfide inclusion which was collected from the fractured fragment of tempered glass quantitatively, because of the fine-grained particle (150 micrometers in mean diameter). The author has introduced quantitative analysis of the degree of beta-phase transformation by micro-Raman spectrometry.

Figure 1 shows the measurement results of both alpha and beta phases of Nickel Sulfide particle included in the molten glass. The beta-phase has four remarkable Raman bands; however, the alpha-phase has no remarkable peaks. We can identify the crystalline phase (alpha or beta) by micro-Raman spectrometry, easily (and speedily).

In general, the Nickel Sulfide inclusion is composed of alpha-beta poly-crystalline phases which contain NiS, Ni₇S₆ and Ni₃S₄ [2]. Therefore, we must identify the individual crystalline phases in the Nickel Sulfide inclusion.

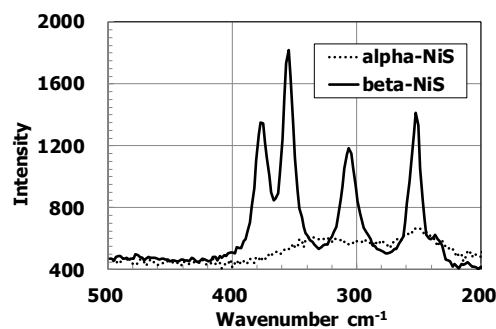


Figure 1. Raman spectra of both alpha and beta Nickel Sulfides

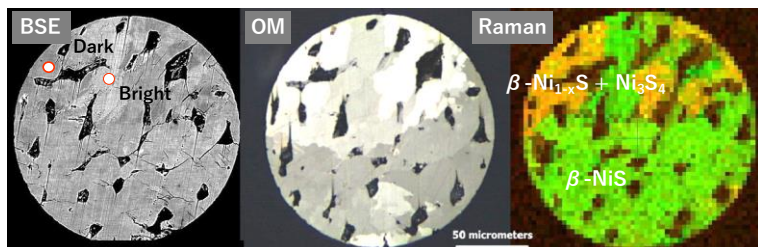


Figure 2. Back Scattered Electron image, optical photomicrograph and micro-Raman image of Nickel Sulfide inclusion

Figure 2 shows the results of analysis of Nickel Sulfide inclusion which was collected from the fractured fragments of tempered sheet glass in the glass market. BSE image shows only chemical change of Ni_xS_y. Optical image (by metallurgical microscope) shows each crystalline domain in the poly-crystalline phase. And, micro-Raman image shows the distribution of crystalline states (alpha or beta) of Nickel Sulfide clearly.

It is difficult to show the quantitative distribution of both alpha and beta of Nickel Sulfide by only metallurgical microscope observation [3]. The inclusion is composed of both "beta-NiS+Ni₃S₄" and "beta-NiS" on the basis of the analysis of micro-Raman image. And, the quantity ratio of each crystalline phase was calculated as follows.

$$\text{beta-NiS} + \text{Ni}_3\text{S}_4 : \text{beta-NiS} = 30.3 : 69.7$$

Therefore, it is considered that the inclusion is composed of 85% or more of beta-phase of Nickel Sulfide. And, the crystallographic calculation shows 2.9% volume expansion after beta-phase transformation in the glass market. Quantitative calculation of both beta-achievement and volume expansion of Nickel Sulfide is very important for the grasp (reliability) of processing performance of Heat Soak Test. And also, it is very useful for the elucidation of breakage cause in the glass market.

References

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