On the measurement of the work-hardening effects of mechanical grinding

Paolo Scardi[1,*], Mirco D'Incau[1]

[1] Department of Civil, Environmental & Mechanical Engineering, University of Trento

38123 via Mesiano 77, Trento, Italy

Abstract. Mechanical milling induces strong work hardening and the formation of nanocrystalline domains in powders, challenging conventional microstructural characterization. This contribution reviews advanced X-ray diffraction line profile analysis methods that exploit the whole powder pattern, providing reliable insight into lattice defects and crystalline domain size. Examples from mechanically milled metals and ceramics illustrate state-of-the-art capabilities.

1. Characterization of mechanically ground powders by X-ray diffraction.

Mechanical milling is known to produce strong work hardening effects and crystalline domain size reduction. This enables the production of highly reactive, finely powdered inorganic materials for a variety of applications, from catalysis to additive manufacturing and advanced powder sintering. In this context, it is essential to understand the effects of grinding, especially under the most extreme conditions, which lead to the formation of nanocrystalline materials.

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are essential tools, but can be limited in observing nanoscale crystalline domains with high defect density and strong agglomeration. Under these conditions, X-ray diffraction (XRD) is particularly useful and appropriate: Line Profile Analysis (LPA) – the study of diffraction peak width and shape – provides information on lattice defects (e.g., stacking faults, dislocations, etc.), and on the shape, size and dispersion of the crystalline domains down to few nanometres.

The most traditional approach to LPA uses only peak widths. Methods based on the Scherrer equation or the Williamson-Hall plot are very popular because they are simple to use, but they rely on crude approximations and provide limited information that can even be misleading [1].

2. Advanced methods for line profile analysis.

Over the last two decades, there has been a significant development in methods that utilize the entire XRD powder pattern information, including all the Bragg peak profiles and the diffuse scattering component. This provides detailed information on the microstructure of materials, particularly if they are nanocrystalline and highly deformed, such as those resulting from mechanical comminution. This new and more reliable approach can be integrated into well-known powder diffraction methods, such as the Rietveld method. Various software programs are available, some of which are commercial and easy to use. [2-4]. This is well known in the XRD community, especially at the level of methodological developments, but reaches less the broad user community, which ranges from basic to applied research in various fields of chemistry, physics and engineering.

This presentation aims to help fill this gap by illustrating the state of the art of LPA methods, with examples applied to nanocrystalline powders obtained by high-energy mechanical milling.

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References

[1] Scardi, P., Leoni, M., Delhez, R. (2004) Line-broadening analysis using integral breadth methods: a critical review. *J. Applied Crystallography* 37, 381-390.

[2] Scardi, P. (2020) Diffraction line profiles in the Rietveld method. *Crystal Growth & Design* 20, 6903-6916.

[3] Scardi, P., Malagutti, M.A. (2024) Thermal Diffuse Scattering from Nanocrystalline Systems. *Crystal Growth & Design* 24, 4380–4392.

[4] Scardi, P., D'Incau, M., Malagutti, M.A., Terban, M., Hinrichsen, B., Fitch, A.N. (2025) A reference material for XRD Line Profile Analysis. *J. Applied Crystallography* 58, 1764-1777.

* Corresponding author: Paolo.Scardi@unitn.it