Multiphase materials with grain sizes in the nano-regime are difficult to characterize structurally, particularly in the as-processed state. XRD observations are made difficult by the extremely broad and overlapping diffraction peaks of low intensity from the various phases. Additionally, observations on a variety of materials, including materials with grain sizes less than about 150 nm, have shown increases in the background intensity when compared to large-grained polycrystalline samples. Analyses of these background scattering effects have usually been qualitative in nature, and although attempts to understand the source of the scattering through simulation methods have provided encouraging results, the simulations typically have discrepancies with the observed background intensities. However, despite a variety of assumed structural forms for the grain boundaries, a common conclusion of most studies has been that the increased background intensity arises from significant defective grain boundary regions.

The current work has observed scattering from an (Al, Cr)_3Ti alloy processed by reactive mechanical milling (RMM) with Ti powder to form an intermetallic alloy matrix with in situ carbide and hydride phases formed during processing. As RMM processing time increases, a variety of interesting changes occur in the observed scattering. These include severe broadening of matrix diffraction peaks accompanied by a significant decrease in maximum peak intensity, appearance of diffraction peaks resulting from the formation of very small crystallites of TiH_{1.92} and TiC phases with increasing volume fractions, and finally, an increasing background intensity with the decrease in the crystallite size of the matrix phase. Detailed analyses provide estimates of phase volume fractions by the direct comparison method, along with crystallite sizes by Warren-Averbach peak profile analysis. The general increase in background intensities has been attributed to random static displacements of the large fraction of atoms located within the grain boundary regions. Combining the approach used by Ruland to estimate the volume fraction of crystallinity within amorphous materials, with the direct comparison method has shown that matrix material with a crystallite size of a few nanometers has about half the atoms in statically displaced positions defining the boundary regions. The results argue that background intensity changes should not be ignored and may be useful in interpreting the scattering from these nano-scale materials.