Optimized in situ electron backscatter diffraction method for continuous heating and its application for studying the recrystallization behavior of aluminum alloys

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Introduction

The study of recrystallization processes is central to understanding material properties after thermomechanical treatment. While *in situ* electron backscatter diffraction (EBSD) has been successfully used for recrystallization characterization for some time [1,2], the conventional approach is to use isothermal annealing or stepwise heating. In this work, a method for *in situ* EBSD using continuous heating at a constant rate is presented. The method is based on optimized acquisition parameters and an improved strategy for evaluating the in situ EBSD scans. Here, the capabilities of the method are demonstrated in recrystallization studies of a cold rolled AA6016 aluminum alloy. The influences of heating rate and previous deformation and also concurrent precipitation on recrystallization are shown and discussed.

Experimental

The EBSD measurements were conducted on a Zeiss Ultra 55 equipped with a Thorlabs Fast Frame Rate Scientific camera and the OIM DC V7.3.1 software. For in situ heating the CH0-4 heating stage from Kammrath & Weiss was used.

Recrystallization shows a very fast transformation in a short temperature interval. Therefore, fast EBSD scans are required to capture the process. Fig. 1 shows the effects of EBSD exposure time when recording the microstructure of cold rolled aluminum alloys in the deformed state (a, b) and in the partially recrystallized state (c). Deformed grains cannot be recorded at low exposure times, while recrystallized grains are well recorded at low exposure times (i.e., fast scans).

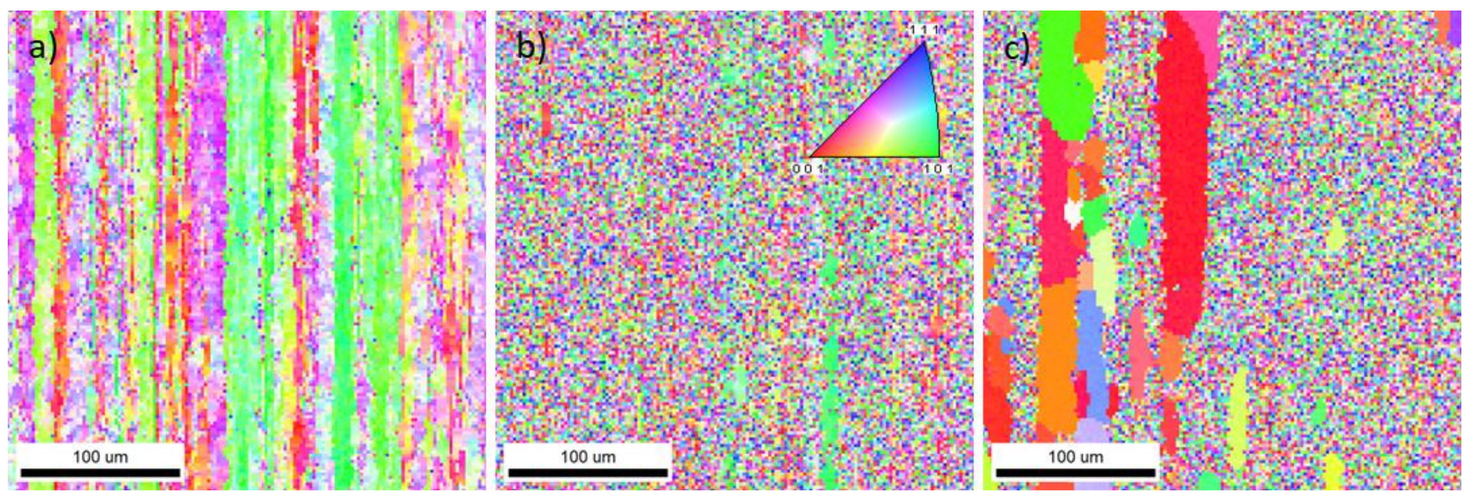

Fig. 1: Influence of exposure time on the EBSD scan:
a) t_e = 20 ms; b) t_e = 2.5 ms; c) t_e = 2.5 ms

Results and Discussion (1)

The evolution of recrystallization during annealing at 4 K/min is shown in Fig. 2. From the *in situ* EBSD maps, the so-called Avrami curves (Fig. 3) were extracted for different heating rates and cold rolling reductions. While the curves for 1, 4 and 7 K/min follow the expected behavior, the recrystallization at 10 K/min takes place at too low temperatures.

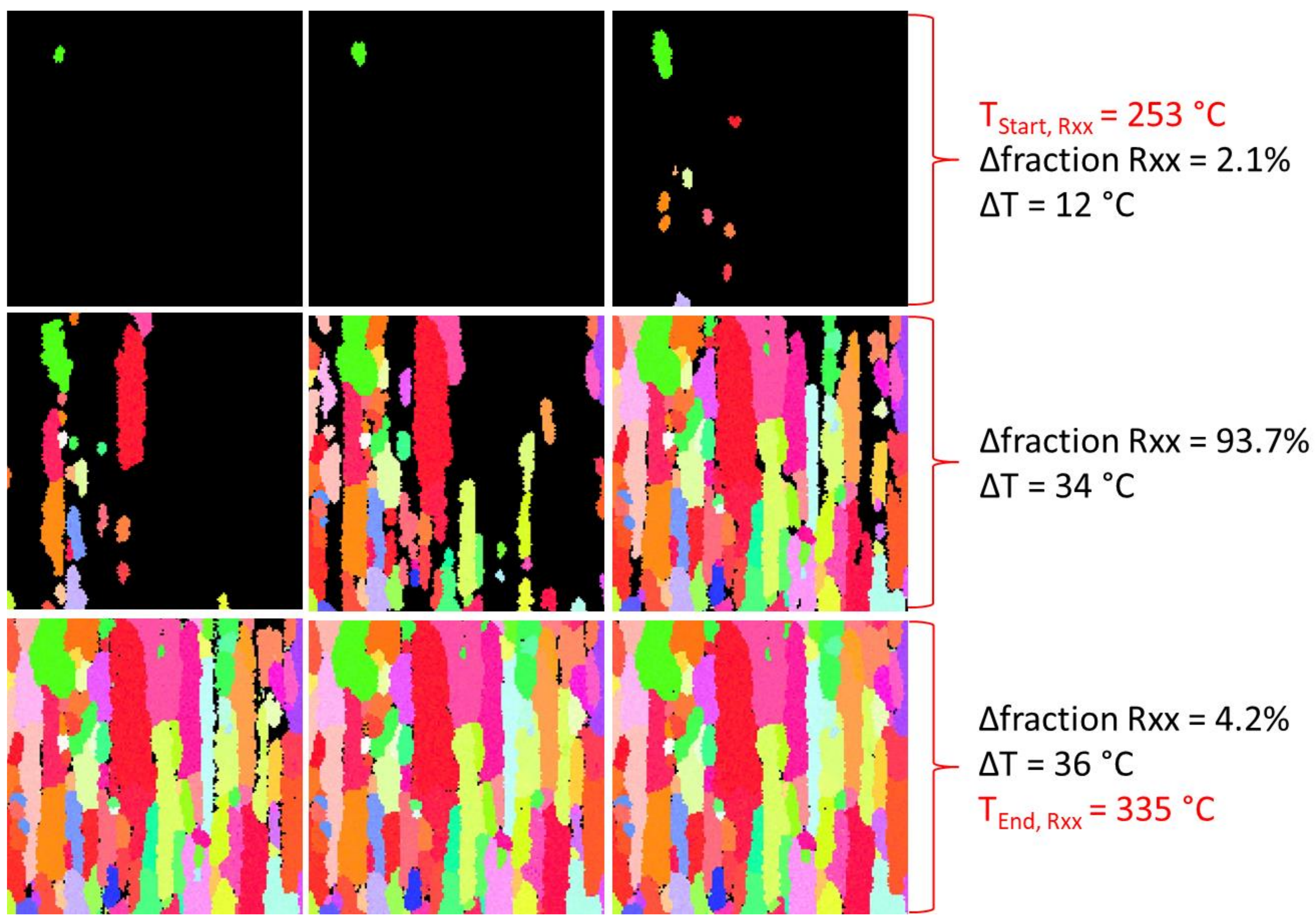

Fig. 2: Evolution of recrystallization during heating with 4 K/min. (sample with 75% cold rolling reduction)

Results and Discussion (2)

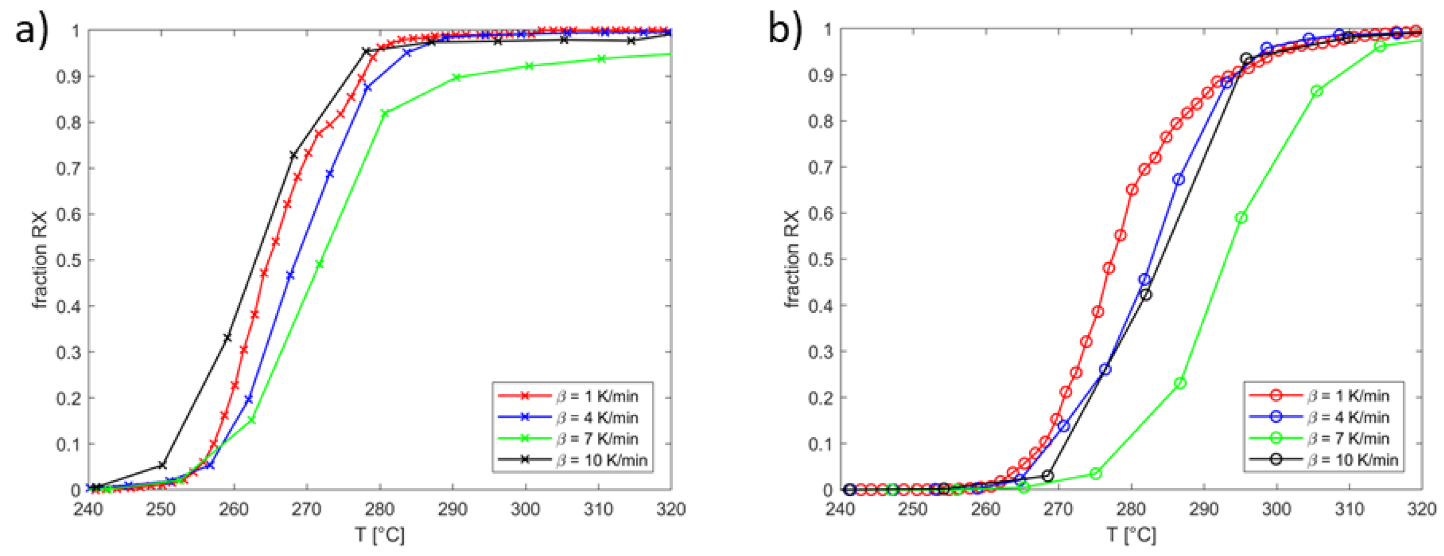

Fig. 3: Avrami curves for different heating rates: a) 87.5% cold rolling reduction, b) 75% cold rolling reduction

One explanation for this is the concurrent precipitation (Fig. 4) which occurs in parallel with the recrystallization process. When heated at 10 K/min, the Mg_2Si precipitates preferentially form at the grain boundaries, i.e., recrystallization precedes precipitation. Therefore, the precipitates are not an obstacle for newly formed grains (low Zener drag). Annealing at 1 K/min allows the Mg_2Si particles to precipitate randomly within the recrystallized grains. This can be interpreted to mean that recrystallization is slower than precipitation, implying that the precipitates are an obstacle to grain boundary movement (high Zener drag). The same precipitation behavior during recrystallization was observed in the sample with higher cold rolling reduction.

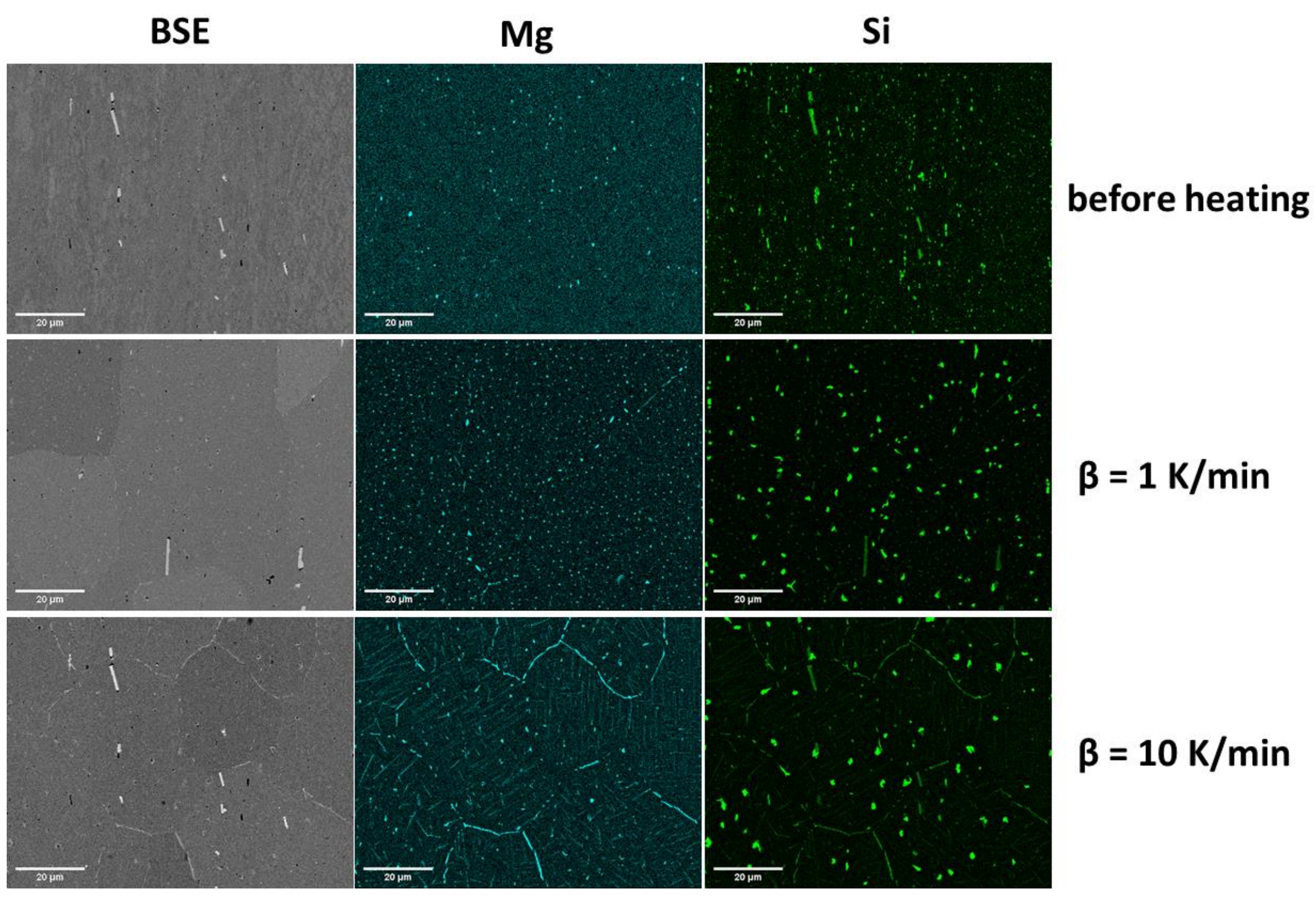

Fig. 4: Precipitation characterization with BSE, Mg K EDX element maps and Si K EDX element maps for different annealing states. (sample with 75% cold rolling reduction)

References/ Literature

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