

Grain growth transitions of perovskite ceramics and its relationship to abnormal grain growth and bimodal microstructures

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Abstract

Barium titanate, strontium titanate and Lithium Lanthanum Titanate (LLTO) were used to study grain growth in perovskite ceramics. In all materials a grain growth transition was found. In the case of barium titanate, grain growth shows a gradual transition to faster growth with increasing temperature whereas strontium titanate indicates a transition region with exponentially reduced grain growth with increasing temperature. In reducing atmosphere, strontium titanate shows a second transition, which is attributed to a reversible wetting transition. In LLTO a single grain growth transition is documented, which seems to be related to a wetting transition as well. In all cases, the transitions are strongly correlated to abnormal grain growth.

The non-Arrhenius behaviour of grain growth in perovskites is discussed in relation to abnormal grain growth and bimodal microstructures. A unified macroscopic grain growth theory is presented, which bases on grain boundary characteristics and the grain boundary anisotropy. Microscopic origins of the grain growth transitions are discussed to complement the macroscopic model. The use of classical mean field models is critically assessed.

Keywords

Grain growth, grain growth transition, grain boundary mobility, abnormal grain growth, strontium titanate, barium titanate, lithium lanthanum titanate

1. Introduction

The evolution of the mean grain size of a microstructure is commonly described by the equation [1-3]

$$r^2 - r_0^2 = \frac{1}{4}kt \quad 1$$

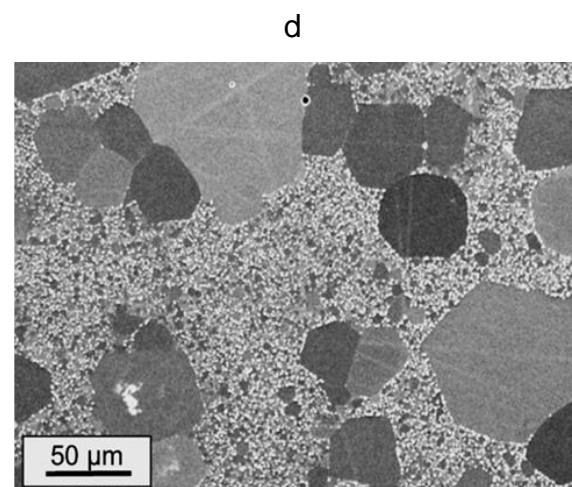
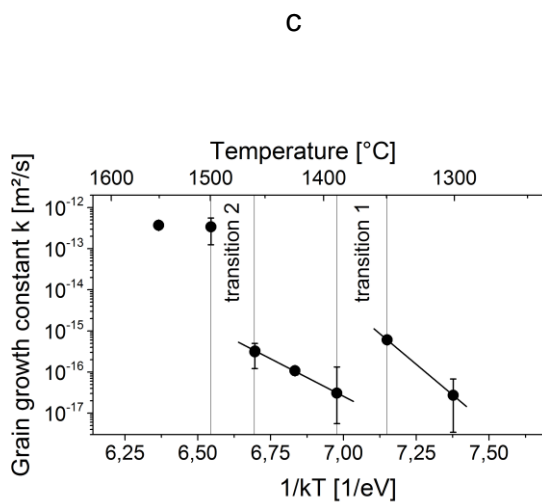
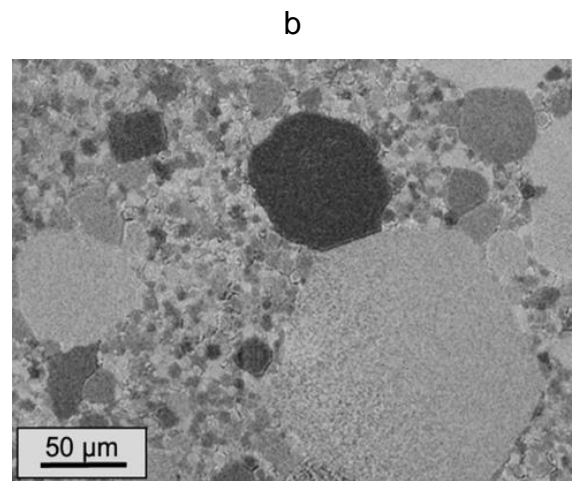
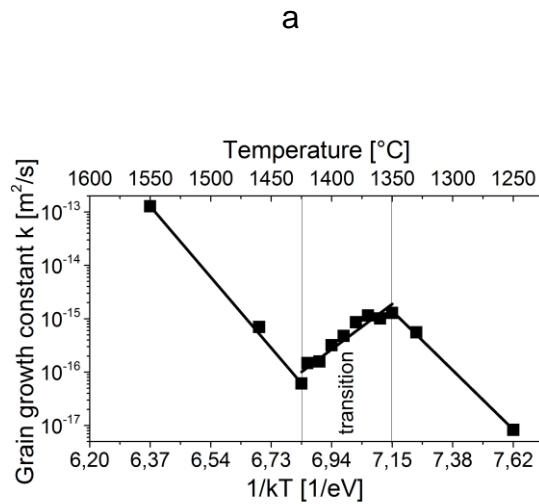
with r as mean grain radius, r_0 as initial mean grain radius at time $t = 0$ and k as grain growth constant. Standard grain growth experiments provide access to the grain growth constant to evaluate the grain growth behavior with respect to temperature. Grain growth is a thermally activated process due to its coupling to diffusion; hence k usually shows an Arrhenius-type behavior. However, several materials deviate from this behavior by showing a transition of the grain growth constant. For example in alumina, segregation (complexion) transitions of the grain boundaries with dopant concentration are known to change the grain boundary mobility and thereby the local grain growth constant by several orders of magnitude [4-6].

However, perovskites are different, since several grain growth transitions are known in high purity materials (i.e. without any influence of dopants). E.g. strontium titanate shows a grain growth transition between 1350°C and 1425°C in oxidizing atmosphere ([7-9], cf. Fig. 1a): with increasing temperature k decreases gradually by almost two orders of magnitude. In conjunction, microstructures are strongly bimodal above the transition temperature (Fig. 1b) with a decreasing fraction of large grains with increasing temperature [9]. In reducing atmosphere, strontium titanate shows a similar behavior with a decreasing k with increasing temperature between 1350°C and 1400°C ([10], Fig. 1c). Additionally, a second grain growth transition to faster growth exists between 1460°C and 1500°C. This transition was related to a grain boundary wetting transition [10] and is again coupled to bimodal microstructures (Fig. 1d).

In barium titanate, a grain growth transition was reported recently as well: an increasing temperature leads to growth being faster than expected from the Arrhenius behavior ([11], Fig. 1e). Again a gradual behavior of the grain growth constant k within the transition is observed. As in strontium titanate, microstructures are bimodal in the temperature range of the grain growth transition (Fig. 1f).

Since microstructures are unimodal at temperatures below the grain growth transition temperature and become bimodal at the transition temperatures [9-11], the grain size

distribution is a very important tool to characterize grain growth transitions. This study delivers new data on the grain size distributions and thereby further supports macroscopic modelling of grain growth transitions in perovskite ceramics. It is argued that the grain growth constant k is not capable of completely reflecting microstructure evolution in the range of the growth transitions. A so far unknown grain growth transition in the perovskite Lithium Lanthanum Titanate (LLTO) is documented, which leads to the assumption that the existence of grain growth transitions seems to be a common feature of perovskite ceramics. In addition to the macroscopic modelling, microscopic origins of grain growth transitions in perovskites are discussed.



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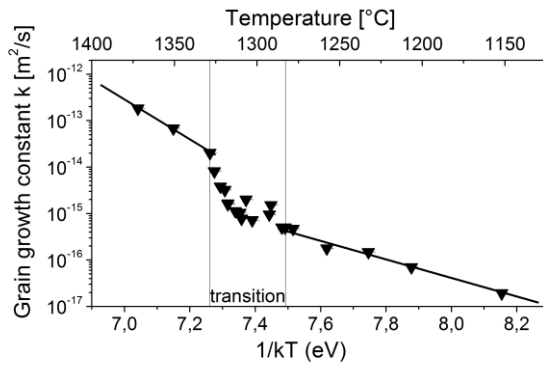


Fig. 1 Grain growth transitions and microstructures in SrTiO_3 in oxidizing (a, b) [9] and reducing atmosphere (c, d) [10]. Grain growth transition and microstructure in BaTiO_3 (e, f) [11]. Within the growth transitions microstructures become bimodal.

2. Experimental Procedure

Stoichiometric strontium titanate powder was prepared using the mixed oxide/carbonate route based on high purity raw materials (SrCO_3 and TiO_2 , Sigma Aldrich Chemie GmbH, Taufkirchen, Germany). The green bodies were sintered at 1425°C for 1h in oxygen to a relative density of $>99\%$ and a mean grain radius of $0.6\ \mu\text{m}$. Further details of the sample preparation are published elsewhere [12]. Lithium lanthanum titanate (LLTO) powder was prepared using the same method, but based on Li_2CO_3 , La_2O_3 (both AlfaAesar, Karlsruhe, Germany) and TiO_2 (Sigma Aldrich Chemie GmbH, Taufkirchen, Germany). The molar Li/La-ratio was 0,43; no presintering was necessary. Samples were quenched to room temperature by initially more than $200\ \text{K/min}$.

Grain growth experiments were done in a tubular furnace (Gero GmbH, Taufkirchen, Germany) in oxygen at ambient pressure. For LLTO, the mean grain radius was obtained by the line intersection method on SEM micrographs. Typically 150 grains were counted (at least 100). The grain growth constant k was obtained by fitting Eqn. 1 to the evolution of the mean grain radius with time.

Grain size distributions were determined using SEM (Nova NanoSem 450, FEI, Hillsboro, USA) and high resolution EBSD (e-Flash HR+, Bruker, Billerica, USA). The step size was below 100nm (typically 75nm); the frame size was 2500×2158 pixels. Typically 7000 grains were quantified (at least 2500). In some cases an EDS analysis

was performed (X-Flash 6|30, Bruker, Billerica, USA). The SEM was operated at 15kV; the EDS measurement time was 30min at ~30kcps.

3. Results and Discussion

3.1. *The role of bimodal microstructures in grain growth transitions of perovskites*

The temperature dependent microstructure evolution in polycrystalline strontium titanate is shown in Fig. 2. All microstructures are bimodal; however from 1390°C to 1425°C the fraction of large grains decreases (i.e. the fraction of small grains increases with increasing temperature). This feature can be quantified best by observing the grain size distributions (Fig. 3): the grain size distribution at 1390°C after 10h shows a very pronounced fraction of large grains with a size larger than 10µm. This fraction decreases drastically at 1400°C after 10h and is negligible at 1425°C after 10h.

Additionally to this pronounced temperature dependence, the bimodality of the microstructures strongly evolves with time. Fig. 4 shows the microstructure evolution at 1390°C after 2h, 5h and 10h. While after 2h only a small fraction of large grains appears, after 10h the microstructure is clearly dominated by large grains. The grain size distributions in Fig. 5 show only few grains with a size above 10µm after 2h and a very large fraction after 10h. As shown by Fig. 6 and 7, at 1400°C the trend is the same as at 1390°C, but as expected according to Figs. 2 and 3, the fraction of abnormal grains is generally lower than at 1390°C.

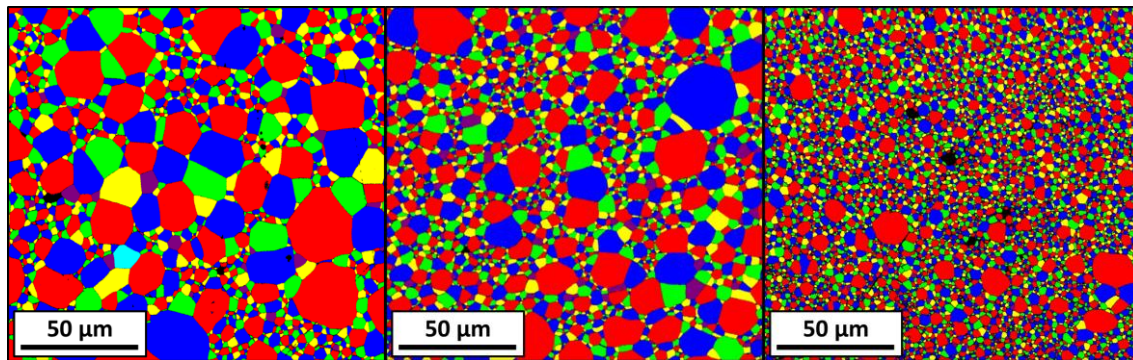


Fig. 2 Microstructures of SrTiO₃ measured by SEM and EBSD after 10h at 1390°C (a), 1400°C (b) and 1425°C (c).

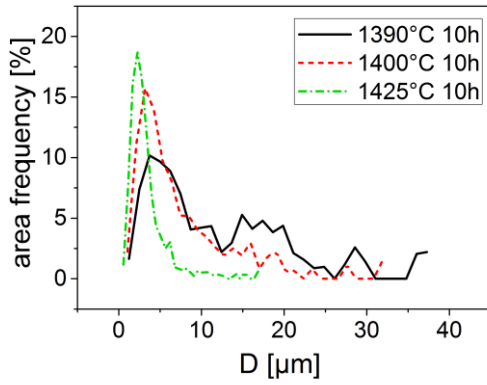


Fig. 3 Grain size distributions after 10h at 1390°C, 1400°C and 1425°C according to Fig. 2a-c.

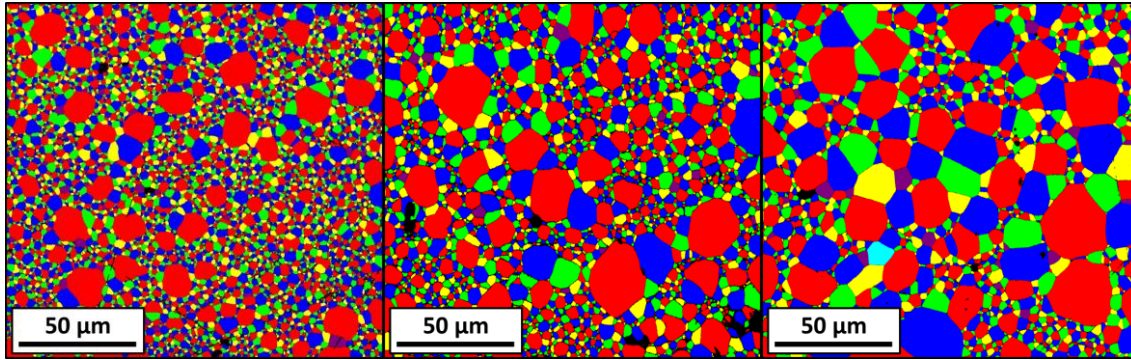


Fig. 4 Microstructures of SrTiO_3 measured by SEM and EBSD at 1390°C after 2h (a), 5h (b) and 10h (c).

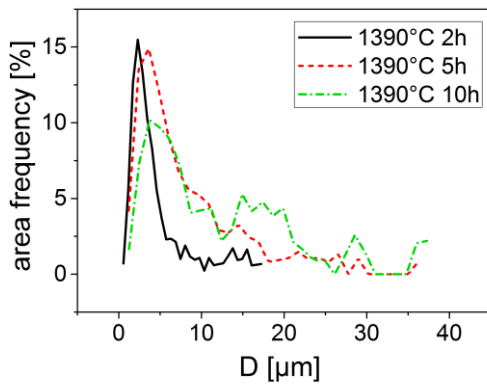


Fig. 5 Grain size distributions at 1390°C after 2h, 5h and 10h according to Fig. 4a-c.

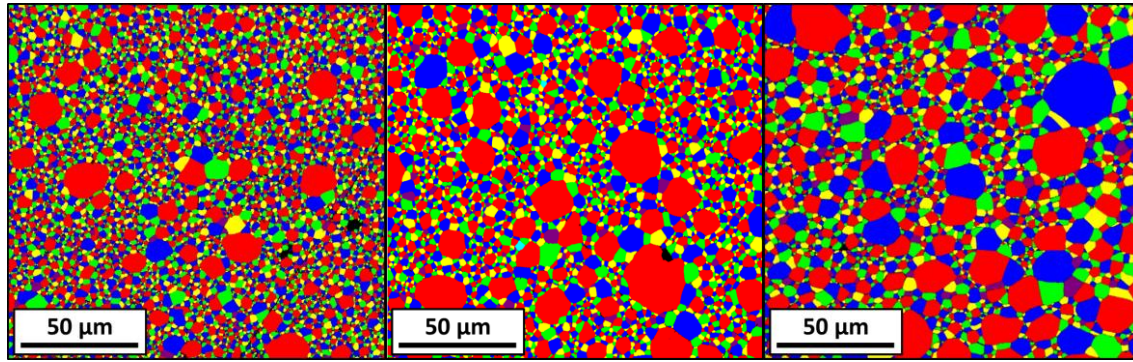


Fig. 6 Microstructures of SrTiO₃ measured by SEM and EBSD at 1400°C after 2h (a), 5h (b) and 10h (c).

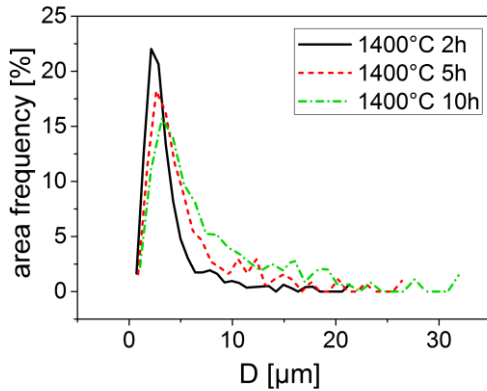


Fig. 7 Grain size distributions at 1400°C after 2h, 5h and 10h according to Fig. 4a-c.

3.2. Grain growth transition in LLTO

The temperature dependent grain growth constant k in LLTO as obtained by observing the evolution of the mean grain size is shown in Fig. 8. In general, the grain growth constant in LLTO is in a similar order of magnitude than in other perovskites [9-11]. Below 1400°C, classical Arrhenius-type behavior is observed. However, above 1400°C a sudden decrease of k by two orders of magnitude was observed. While below 1400°C microstructures are unimodal (Fig. 9a), from the transition temperature of 1400°C on microstructures are bimodal (Fig. 9b-d). Thus LLTO shows a grain growth transition in conjunction with bimodal microstructures similar to other perovskites (Fig. 1).

Above 1425°C, a wetting second phase film was found at the grain boundaries (white arrows in Fig. 10a and b). An EDX mapping was applied to a thick region of the wetting second phase. According to Fig. 11 the wetting second phase is Ti-rich. Its composition

was found to be 69.55 at-% O, 28.66 at-% Ti, 1.43 at-% La and 0.35 at-% Zr. The Zr originates in the powder processing (zirconia abrasion during milling [12]). No other elements were found; Li is not detectable by EDS.

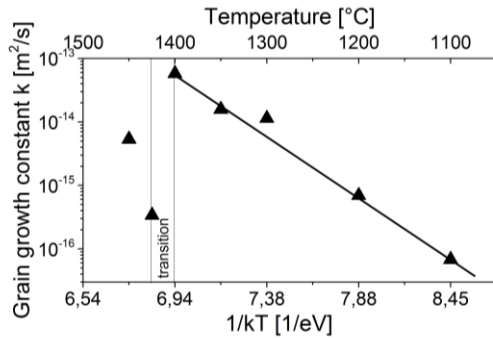


Fig. 8 Grain growth constant of LLTO. Between 1400°C and 1425°C a grain growth transition occurs.

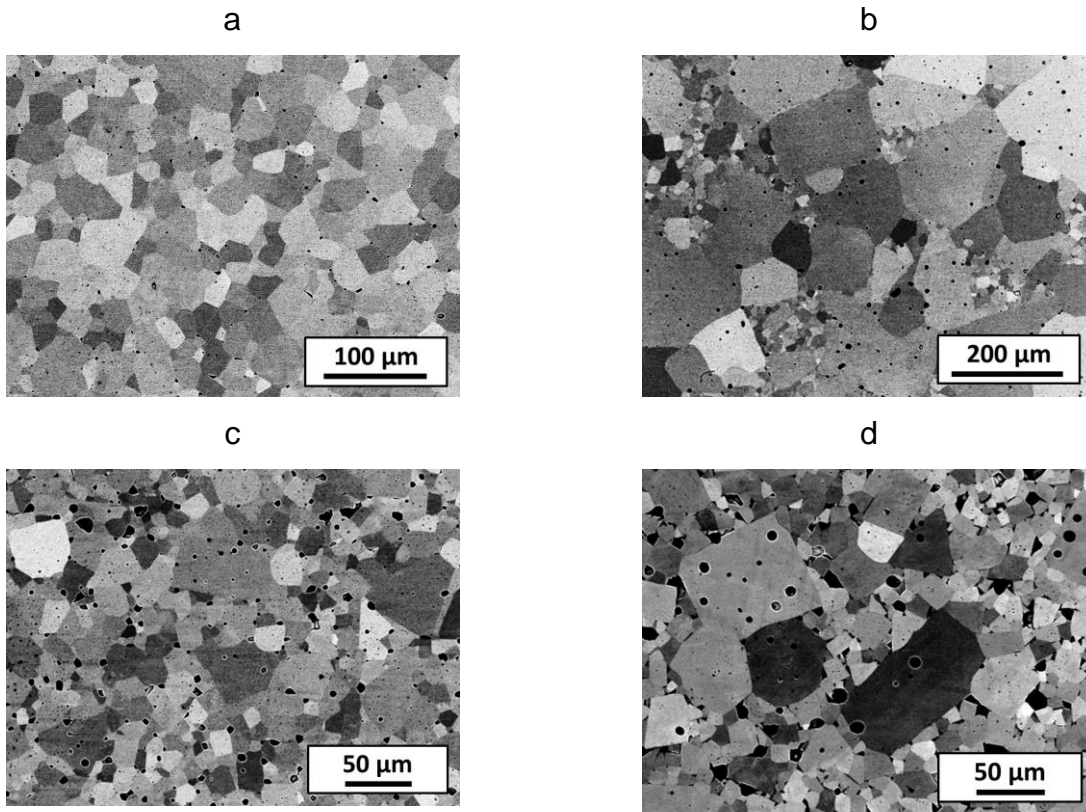


Fig. 9 Unimodal microstructure in LLTO at 1350°C after 10h (a). Bimodal microstructures in LLTO at 1400°C after 10h (a), at 1425°C after 5h (b) and at 1450°C after 5h (c).

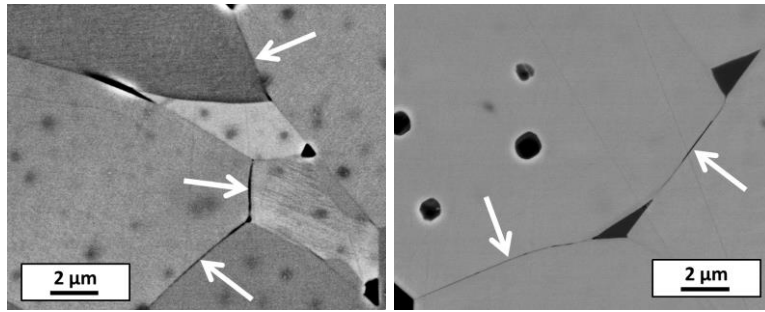


Fig. 10 Microstructure of LLTO at 1425°C after 5h (a) and at 1450°C after 5h (b). White arrows highlight a wetting second phase.

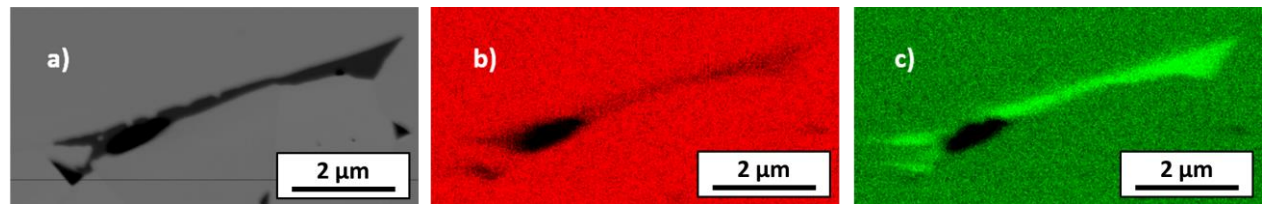


Fig. 11 SEM image (a) and distribution of La (b) and Ti (c) at the wetting second phase in LLTO at 1450°C after 5h as measured by EDS.

3.3. *Macroscopic theory for grain growth transitions in perovskites*

Although certainly of different microscopic origin, the grain growth transitions of the perovskites SrTiO_3 , BaTiO_3 and LLTO can be explained by the same macroscopic model, which proposes the existence (and coexistence) of two grain boundary types. The two types indicate a different grain boundary mobility [9]: Type 1 has a high grain boundary mobility and type 2 a low grain boundary mobility. If these two grain boundary types coexist, the fraction of fast type 1 grain boundaries results in a fraction of large grains in the microstructure, while the fraction of slow type 2 grain boundaries results in a fraction of small grains. Accordingly, this coexistence necessarily results in bimodal microstructures, which were found to be characteristic for the grain growth transitions of perovskite ceramics (Para. 3.1, Fig. 1, Fig. 8) and were found in a similar situation for alumina as well [4]. This assumption is well-supported by the growth rate of the fraction of large grains in strontium titanate, which follows Arrhenius-type behavior of the fast type 1 grain growth [9].

The grain growth transitions found in SrTiO_3 , BaTiO_3 and LLTO now simply represent a change of the initial fractions of type 1 and type 2 boundaries in the microstructures: a decreasing growth constant with temperature is caused by an increasing fraction of slow type 2 boundaries in the microstructure (SrTiO_3 in oxidizing atmosphere and LLTO), an increasing grain growth constant is caused by an increasing fraction of fast type 1 boundaries (SrTiO_3 in reducing atmosphere and BaTiO_3). The gradual character of the transitions found in strontium titanate [13] and barium titanate [11] could be caused by the anisotropy of the grain boundary energy. In general, a grain boundary will show a transition of its structure, if the grain boundary energy is lowered [5, 6]. The grain boundary energy of perovskites is anisotropic [10, 13-15]; accordingly the transition temperature of individual boundaries may vary. For example if a wetting transition occurs, high energy grain boundaries should transit at lower temperatures than low energy grain boundaries [10]. Hence the gradual transition of the total grain boundary area from one type to another and, thus, of the grain boundary rate could be a consequence of the grain boundary energy being anisotropic.

However, the grain growth constant k is not capable of accurately reflecting the growth of bimodal microstructures: if grain boundaries of type 1 and type 2 coexist, its fractions in the microstructure cannot be constant with time. This is obvious in Fig. 4 and Fig. 6. In both cases, the microstructures indicate a very small fraction of large type 1 grains after 2h. This fraction increases with time, until after 10h at 1390°C very few type 2 grains are left and the microstructure is dominated by large type 1 grains. In this case the use of a mean grain size results in an error in the grain growth constant: k always resembles the growth of the dominating fraction of grains in the microstructure, which are small type 2 grains for very short dwell times and large type 1 grains for sufficient long dwell times. As long as both populations represent reasonable fractions in the microstructure, the k obtained by the overall mean grain size is between these two extremes. Accordingly, in bimodal microstructures it is necessary to distinguish between the growth constant of small and large grains. However, in SrTiO_3 , the two populations are not clearly separated in the grain size distributions shown in Figs. 3, 5 and 7. This could be caused by the anisotropy of the grain boundary mobility [10, 16]: each grain boundary type does most probably not have a single mobility, but rather a range due to the grain boundary

anisotropy. Accordingly, numerical tools as anisotropic grain growth simulations are needed to accurately characterize bimodal grain growth in perovskite ceramics.

The macroscopic model of two grain boundary types is in very good agreement with the microstructure evolution of SrTiO_3 , BaTiO_3 and LLTO, accordingly it seems as grain growth transitions are a common feature of perovskite ceramics. However, no detailed grain growth data exists for other perovskites. But bimodal microstructures are characteristic for grain growth transitions and many perovskites are known to show bimodal microstructures, e.g. KNN [17-20], NBT [21, 22] and PMN-PT [23, 24]. Thus the existence of grain growth transitions in these materials is likely, although abnormal grain growth can not only result of two different boundary types, but as well of completely different growth mechanisms such as twinning [25, 26]. Further experiments are needed to examine grain growth and to confirm the existence of grain growth transitions in these materials.

3.4. Microscopic origins for grain growth transitions in perovskites

At atomic scale, the origin of the grain growth transitions of perovskites is very different depending on the material and is not known in detail in all cases. E.g. for BaTiO_3 (Fig. 1e) few information is available, although the occurrence of a thermal roughening transition of the boundaries is most probable [11, 14, 27, 28]. This assumption is supported by the change in activation energy across the transition, which does not occur in SrTiO_3 (Fig. 1a and c).

For strontium titanate in oxidizing temperature (Fig. 1a), the grain growth transition between 1350°C and 1425°C was found to be correlated to a change in the grain boundary anisotropy [13]: The grain boundary plane distribution (GBPD) indicates an increasing fraction of low energy planes (primarily $\{100\}$) with increasing temperatures. At higher temperatures $\{100\}$ is a low mobility orientation [16]. Grain growth simulations show that even a small fraction of low mobility GBs decreases the grain growth constant considerably [11, 29]. Accordingly the increasing frequency of grain boundary planes oriented in $\{100\}$ with temperature seems to be related to the grain growth transition of strontium titanate in oxidizing atmosphere [13, 16, 30]. On atomic scale, no structural change of the grain boundary could be found by TEM [31]. However, analytical TEM indicates the trend that Ti-rich GBs are slower than others [32]. However, the details of

the correlation to the anisotropy of the grain boundary mobility and the grain growth transition are not clear so far.

For strontium titanate in reducing temperature, a second grain growth transition at higher temperatures (1460°C) was documented recently [10]; it seems to be caused by a reversible wetting transition: above 1460°C grain boundaries were wetted by a Ti-rich second phase and, thus, showed a high grain boundary mobility. Bimodal microstructures seem to be caused by the anisotropy of the wetting transition [10]. According to Para. 3.2, the grain growth transition in LLTO (Fig. 8) seems to be caused by a wetting transition as well. However, in contrast to strontium titanate, wetted boundaries have a lower mobility than non-wetted boundaries and the transition is inverse to the transition of strontium titanate.

4. Summary and Conclusion

This study shows that grain growth transitions seem to be a common feature in perovskite ceramics. The grain growth transitions result in a non-Arrhenius behavior of the grain growth constant k , which shows transitions to slower (SrTiO_3 in oxidizing atmosphere and LLTO) or faster grain growth (SrTiO_3 in reducing atmosphere and BaTiO_3). In all cases a pronounced bimodality of the microstructures was found in the temperature range of the transition.

Macroscopically, the grain growth transition can be explained by the existence and coexistence of two different grain boundary types, which indicate different mobilities. A coexistence of these types results in the observed bimodal microstructures. The growth transitions are caused by a gradual change of the grain boundary area fractions of the two boundary types. The change of the area fraction is proposed to be caused by the anisotropy of the grain boundary energy.

However, the area fraction of the two grain boundary types was shown not to be constant with time: during growth, the fraction of fast grain boundaries increases. Accordingly, classical grain growth modelling and the use of the grain growth constant k cannot accurately describe grain growth in perovskite ceramics; anisotropic grain growth simulations are needed to investigate the grain growth transition more in detail.

Microscopic origins for the grain growth transitions are discussed. In BaTiO_3 , the transition is most probably caused by thermal roughening. In SrTiO_3 in oxidizing

atmosphere, the anisotropy of the boundaries (i.e. the temperature dependent frequency and mobility of the orientation {100}) was found to be important. In SrTiO₃ in reducing atmosphere and in LLTO, a wetting transition seems to cause the grain growth transition.

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