

Polycrystalline ceramics: sub grain structure



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Pure un-doped zirconia is a polymorph which has three allotropes namely: Monoclinic, Tetragonal and finally Cubic. These phases tend to transform into each other when exposed to certain temperature ranges and such transformation is important for the processing and mechanical properties of zirconia. The monoclinic phase of pure un-doped zirconia is stable at room temperature and remains so up to about 11700C, where it then transforms into tetragonal phase. It becomes stable tetragonal at this temperature and remains so up to 23700C, where it turns to cubic. The cubic phase occurs up to the melting temperature of 26800C.

Monoclinic phase

The monoclinic form also referred to as baddeleyite, is a thermodynamically stable phase at a temperature range between room temperature and approximately 9500C. It contains four ZrO₂ molecules per unit cell and has a space group of P21/c. Figure 2. 1 shows the lattice parameter of monoclinic form. Its structure is described as a distorted fluorite (CaF₂ structure). It is difficult to define the crystal structure of monoclinic zirconia because of its complexity as well as the problem of making a monoclinic single crystal with the satisfactory qualities due to: micro-cracking, low purity, twinning and disproportionate solid solution formation.

Tetragonal phase

This is a high temperature phase (t) firstly discovered by a group of scientist during its transformation from the lower temperature monoclinic phase over a temperature of about 11500C. Figure 2. 1 shows the lattice parameter of tetragonal form. The structure is similar to that of monoclinic polymorph in the sense that it is also distorted CaF₂ structure. Hence, tetragonal zirconia

(t-ZrO₂) can be described using the face centred tetragonal Bravais lattice as oppose to the body centred tetragonal lattice, that contains a unit cell with volume twice the size of the primitive cell. (3) Figure 2. 2b shows a simple schematic of a tetragonal unit cell. Its structure comprises of eight oxygen ions surrounding a zirconium ion, with half at a distance of 0. 2455nm forming an elongated tetrahedron and the remaining four are at a distance of 0. 2065 forming a flattened tetrahedron (the elongated and flattened tetrahedron are rotated 90° to each other). The transformation from tetragonal to monoclinic can start (T_s) and finish (T_f) over a range of temperatures. This reaction can be measured using the following experimental techniques: DTA, XRD and dilatometry. (3)

Cubic phase

Unlike the other structures, the cubic polymorph is quite easy to explain as it has a fluorite structure (CaF₂). Figure 2. 2c shows a simple schematic of a tetragonal unit cell. It has a lattice parameter of the order 0. 508nm (this however depends on the temperature purity of zirconia that is partially stabilised zirconia at room temperature or pure zirconia at elevated temperature) and a crystal symmetry of Fm3m.

The martensitic transformation

For a martensitic transformation to occur, a change in shape is required which must also produce a plane that does not change during transformation. This is so that it is common to the phase produced as well as the parent phase. The phase transformation in zirconia involves a change in volume of between 4 to 5%. The matrix inhibits the transformed particle of zirconia causing a partial shape change. However, the transformation

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creates a strain which is held in the monoclinic and its surrounding grains. As a result of this, researchers have come up with the idea that transformation stresses are relieved by deformation twinning. When this happens, most of the lattice strain is then restricted to the monoclinic/matrix interface. Micro-cracks can be formed at this matrix/monolithic interface or in the monoclinic particle if this lattice strain increases. The twinning found in monoclinic is caused by deformation twinning, as the researchers have observed using TEM that a section of the strain related with the transformation happens as a result of a mechanism known as slip. (3)

The phase transformation particularly from tetragonal to monoclinic is of great importance, as it attributes the zirconia's excellent properties. [from fulltext. pdf] It was firstly discovered by Garvie et al that the transformation of metastable tetragonal phase to monoclinic phase acts as a toughening mechanism to crack propagation resistance in zirconia. The transformation is quick and results in a 4 to 5 percent increase in volume which leads to formation of micro-cracks and eventually macro-cracks in the material. This process induces compressive stresses and thus toughens the materials.

Gupta et al backed this theory up. Studies showed that the transformation mechanism is highly dependent on grain size and by doping the ceramic material with stabilisers. Examples of stabilisers are yttria (Y₂O₃), magnesia (MgO), calcia (CaO), etc. Y-TZP ceramics is in the family of these toughened materials. Tetragonal zirconia doped with Yttria (Y-TZP) has great strength of over 1000MPa and toughness weighing between 6 and 10 MPa. m^{1/2}. This makes it an ideal contender in medical applications, particularly in hip joints.

ZrO₂ - Y₂O₃

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The phase diagram shown in figure 3 was firstly discovered by Scott (1975), this study was agreed and used by many more researchers. The tetragonal phase field is the main aspect of figure 3. It shows that up to about 2.5 mol% of Ytria can be produced in solid solution in addition with the low eutectoid temperature leading to the formation of a fully tetragonal ceramic, this will happen as long as the grain is of an appropriate size.

The theory of transformation toughening produced some excitement in the materials industry however this excitement came to a halt when Kobayashi et al discovered a flaw in Y-TZP ceramics. Y-TZPs undergoes low temperature degradation during ageing at temperatures ranging from 100 to 4000C, this is particularly enhanced when it is exposed to water or is in humid environments. This degradation is due to the formation of flaws such as micro-cracks and macro-cracks (mentioned earlier) at the surface which gradually goes into the bulk of the material. These flaws are due to the spontaneous transformation from tetragonal phase to monoclinic phase.

Material scientists have documented literature regarding the degradation however there have been contradictory views as to the mechanism of this phenomenon. Figure 4 is a graph showing the low temperature degradation of different types of TZPs. Figure 5 shows ageing temperature against surface monoclinic levels. Some of these researchers focused on the interaction between water (or water vapour) and YTZP, whilst others focused on ways to prevent this from happening.

Sato et al came up with a theory where the hydroxyl group from water (H₂O) reacts with zirconia from the bonds between zirconia and oxygen (that is Zr-

O-Zr bonds) forming Zr-OH bonds at crack tips. This accelerates the rate at which the metastable tetragonal phase transforms to monoclinic at low temperatures. They came up with the conclusion that there is a strain which stabilizes the tetragonal phase, however under certain circumstances it is released and with the combination other pre existing flaws accelerates the transformation.

The theory put forward by Yoshimura et al is similar to that of Sato et al in the sense that the Zr-OH bonds are also formed. However, the reaction process which leads to the same outcome is what differentiates the two theories. Their research showed a comparison of the transformed monoclinic phase to the untransformed tetragonal ZrO₂. Hydroxyl (OH⁻) was in the monoclinic ZrO₂ whereas there was no trace in the latter. Due to their findings, they came up with the theory that the degradation process occurred in stages: upon exposure to water, Zr-OH bonds are formed as a result of H₂O being adsorbed on the YTZP surface. This creates a stress site which builds up as the OH⁻ ions diffuse through the surface and lattice causing the formation of nucleation sites for the phase transformation. This occurs until the stress reaches crack level causing the transformation to occur at the surface leading to the formation of micro and macro cracks all the way through to the bulk.

Lange et al [7] witnessed $\hat{I}\pm$ -Y(OH)₃ crystallites of about 20 – 50 nm in size forming and came up with the idea that the hydroxide formed creates a monoclinic nuclei by removing Ytria from the grains of the tetragonal phase on the surface. As Ytria is being withdrawn, growth of the nuclei continues until a critical size where it will grow spontaneously, leading to the

transformation of tetragonal grains to monoclinic. Micro cracks and macro-cracks begin to occur as the transformed grain gets large enough. This process happens over and over again as the micro and macro-cracks act as a site for water molecules to penetrate into to the grains. This process occurs only if the grains are larger than the critical size. However, if they are smaller, the transformation will be influenced by the diffusion of Yttria on the surface. Other researchers such as Winnubst and Burggraf support this theory, as they found traces of Yttria on surface layer of the YTZP specimen. Their specimen was exposed to temperature of 1770C in a nitrogen environment for over 5hrs and using an auger electron microscope, they found a yttrium rich surface layer.

The listed theories were based on YTZP's mechanism during degradation. Whalen et al identified that the reason for this degradation is the spontaneous transformation from tetragonal phase to monoclinic phase at the surface which then eventually spreads to the bulk. They came up with the idea of stabilising the tetragonal phase. This could be done by either of the following two methods: the chemical factor which is increasing the stabiliser content on the surface or the microstructural solution which is reducing the grain size at the surface. The latter was decided upon and this was done by the process of post sintering grinding followed by annealing treatment.

2. 45mol% Y₂O₃/ZrO₂ was the material involved in the research. Samples of the material were made using isostatic pressing at pressure of 275MPa and then sintered at a temperature of 1500C for a time period of 2hrs. A 2mm disk was formed of which its two sides had different surfaces treatments,

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One side being polished and the other being surface grounded. The phase compositions at surfaces were then examined using XRD. The XRD result indicated there was a significant difference in the phase composition of both sides. The ground side showed little transformation change whereas there was 50% increase in monoclinic phase after annealing. This provided evidence that the ground and annealed surface hindered the process of phase transformation from tetragonal to monoclinic at the surface. As a result of this, there were no micro-cracks formed at the surface and hence the expected mechanical properties were achieved.

Talk and compare it to mine later (TEM as oppose to XRD, advantages of process)

The aim of this project is to provide evidence (if any) of the occurrence of refined grains (recrystallization) in Y-TZP structures as a result of deformation. The ideology used to explain the concept of recrystallization in metals can be used to explain its occurrence in ceramics as this is a new phenomena in the ceramic industry.

Grain refinement requires certain conditions in its exposure in polycrystalline ceramics and they are: plastically deforming the material (as a result of applying a stress) and followed by heat treatment.

Deformation is basically a change in body shape which occurs as a result of an applied force. Materials may experience either elastic which is impermanent deformation that upon the release of an applied stress is recovered or plastic deformation which is permanent deformation that is non recoverable when a stress is applied. YTZP's recrystallization behaviour can <https://assignbuster.com/polycrystalline-ceramics-sub-grain-structure/>

be explained by its ability to plastically deform. The stress and strain behaviour of a material is used to determine the start and the degree of plastic deformation.

Figure 6 shows an example of a typical stress and strain curve. Yield tensile strength is the point at which elastic deformation ends and the material begins to plastically deformation. Most polymers and metals undergo elastic followed by plastic deformation but this is not the case for ceramics. They undergo elastic deformation followed by fracture with little or no plastic deformation. YTZP has superplasticity properties and this nature can be used to explain refinement in its microstructure.

Plastic deformation is governed by the movement of large numbers of dislocations. Hindering dislocation motion will increase a material's strength. Ceramics are inorganic materials held together by both ionic and covalent bonds. The bonding combination results in hindering the motion of dislocations, hence their high strength but brittle behaviour.

Dislocation is an important factor in understanding plastic deformation and so certain elements need to be examined in order to understand the concept. Most materials comprise of an arrangement of atoms referred to as a crystal structure (these can either be single or polycrystalline that is having multiple crystals as the name suggests). This project will focus on polycrystalline zirconia, however understanding single crystals help in explaining the behaviour of polycrystalline materials. All crystal structures have flaws that distort the regular arrangement of the atoms. These flaws can either be point defect (that is they may have vacancies or interstitials),

surface, line (dislocations) and volume defects. The activities and effects of all these flaws are interconnected thus the importance in the need to understand them.

As the dislocations move, they tend to interact with one another however this interaction is a complex as an amount of dislocations (rephrased from pdf). The collective motion of dislocations leads to gross plastic deformation.

<http://composite.about.com/library/PR/2001/blmpi1.htm>

Dislocations can either be screw, edge or a hybrid of both.

Edge dislocation: in this dislocation, the line of defect is parallel to the shear stress. The dislocation movement is similar to that of a caterpillar in the sense that the motion is in small amounts at a time. Figure 7 shows a typical schematic of the motion of dislocations. A is the extra half plane of atoms. As shear stress is applied, the bond between the upper and lower part of B is broken. The extra atom plane of atom A bonds with the lower part of B converting the lower part to an extra half plane. This motion causes the top half to move with respect to the bottom half.

Screw dislocation: this is similar to that of edge in the sense that it also occurs with shear stress however, the defect line is perpendicular to the shear stress as oppose to being parallel. Just like the edge dislocation only a minute fraction of bonds are broken at a given time.

Although the motions are different, the overall plastic deformation for both dislocations is the same. The primary mechanism that causes plastic

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deformation in crystals is called slip. As dislocations move across the crystals, they shear the crystals along their planes of motion.

Slip System

The degree of ease of motion of dislocations is different with in all crystallographic directions and crystallographic planes of atoms. Normally dislocation motion occurs in a preferred plane and within that plane there are specific directions at also which it occurs. The combination of the plane and direction is referred to as a slip system. The plane at which this motion occurs is referred to as slip plane, and the direction is referred to as slip direction. The slip system depends on the crystal structure of the material. Slip will only occur when the value of applied the shear stress exceeds a certain critical value. The mechanism at which slip occurs is different in single crystals that of polycrystalline materials. Schmid defined the critical shear stress in single crystals as shown in figure 9:

Deformation is much more complicated in polycrystalline materials as the crystallography orientations of numerous grains have to be taken into account. This orientation is random and therefore causes the direction of slip to vary from one grain to another. Its complexity extends further more to the grain boundaries which acts as barriers to dislocation motion.

Twinning is another mechanism at which plastic deformation can occur. The idea of twinning in plastic deformation is to allow further slip to occur by producing changes in plane orientations. It occurs when a fraction of the crystals adopts an orientation that is correlated to the orientation of the rest

of the untwined lattice in an exact proportioned way. Figure 10 shows an illustration of an un-deformed crystal with one undergoing slip and twinning.

There is a clear difference between slip and twinning. The crystal orientation in a slip is the same above and below the slip plane whereas in twinning differs across the twin plane. More differences is illustrated in figure 11

Slip

Twinning

Where it occurs

Widely spread planes

Every plane of region is involved

Occurrence

On many multiple slip systems simultaneously

On a particular plane for each crystal

Time required

Milli seconds

Micro seconds

Size (in terms of inter atomic distance)

Multiples

Fractions.

ANNEALING PROCESS LEADING TO RECOVERY, RECRYSTALLIZATION AND GRAIN GROWTH

Annealing is a high temperature process that causes changes in a material's structure, leading to alterations in its properties. When a material is plastically deformed, majority of the energy is dissipated as heat, but a minute fraction is stored in the material as strain energy which is associated with a range of lattice imperfections established as a result of deformation. The deformation process as well as a number of various factors (such as temperature and rate of deformation) determines the amount of energy stored in the material. A reduction in deformation and an increase in intensity of deformation cause a vast increase in the amount of retained energy.

The release of stored energy

There are two main techniques of releasing the energy retained by a material due to plastic deformation and they are an-isothermal annealing and isothermal annealing. Anisothermal annealing occurs when the material is continuously heated from a lower temperature to that of a higher one (the energy discharged is determined as a function of temperature) whereas, Isothermal annealing occurs when the temperature is constant.

The material's microstructure will undergo either or maybe all of these three restoration processes: recovery, recrystallization and grain growth. The extent of plastic deformation can sometimes determine the mechanisms of

recovery and recrystallization. These processes require heat treatment to cause rearrangement of grain boundaries and dislocations.

Recovery

It is the initial stage of annealing that takes place at the low temperature stage of annealing. As a material is plastically deformed, a minute portion of mechanical energy is stored which exists in crystals as stacking faults, point defects (such defects are interstitials and vacancies) and dislocations. When a material is plastically deformed, it is at a thermodynamically unstable state of higher energy. This is converted to lower energy states by the application of annealing leading to a change in microstructure.

There are two process involved in recovery: slip annihilating and polygonization. Slip annihilation occurs when dislocations of opposite signs (that is in the case of edge dislocations, the fusion of the positive and the negative edge dislocation or in the case of screw in which the right hand screw merges with the left hand screw) merge together thereby cancelling each other out. Polygonization is the rearrangement of dislocation after annihilation recovery to a lower energy configuration.

During recovery, this strain energy built up is relieved to some extent by dislocation motion, due to enhanced atomic diffusion at high temperatures.

Recovery leads to physical properties like thermal and electrical conductivities being recovered to their pre worked states. [ggbk]

Recrystallization

After recovery, grains are not entirely strain free. That is the energy state of the grains is relatively high. New sets of strain free grains having near equal

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dimensions in all directions with low dislocation densities are formed. This process is known as recrystallization. This mechanism of producing new equiaxed grains is driven by the difference in internal energy between the unstrained and strained material. The process of recrystallization can occur after or during deformation. The manner at which recrystallization occurs is of two kinds which vary with materials. Firstly a continuous manner, at which the microstructure gradually evolves into a recrystallized one or a discontinuous manner at which distinct new grains nucleate and grow. Recrystallization after deformation is referred to as static whereas the latter is known as dynamic.

The extent at which recrystallization occurs is dependent on two factors namely: time and recrystallization temperature. The temperature at which recrystallization is completed in an hour is referred to as recrystallization temperature. It is usually a third to half the materials melting temperature. The rate at which recovery process occurs is inversely proportional to time (that is it reduces with increasing time). Recrystallization has an entirely different kinetic. During the isothermal annealing, recrystallization starts very slowly then builds up gradually up to a certain point where it slows down. This can be shown in figure 13

In some cases it can be as high 0.7th the melting temperature. An illustration of the relationship between recrystallization temperature and percentage cold work is shown in figure 14. It is understood that as the percentage cold work increases, the recrystallization temperature decreases.

Other factors affect the rate and occurrence of recrystallization. The annealing temperature is one of a few factors that have an effect on recrystallization. A materials recrystallization temperature reduces annealing time. The stress applied is another factor both recrystallization and temperature, an increase in stress applied means a lower temperature is required to activate the process. Also, the deformation on the material must be enough to allow nucleation and growth.

A process known as grain growth occurs in a polycrystalline material after recrystallization provided the annealing temperature is maintained. The restoration mechanism does not require prior deformation or recrystallization and therefore will occur during annealing in their absence in a polycrystalline material. Grain boundary is the driving force for recovery.

Stored energy produced as a result of a material being plastically deformed is released during the process of annealing causing a change in microstructure. This energy released is as a result of various mechanisms due to crystal defect interactions:

A decrease in crystal defects due to their reactions with each other.

Dislocations with opposite signs interacting causing their annihilation and dislocation loop shrinkage.

Relocation of dislocations causing the formation of lower energy configurations such as grain boundaries with low angles.

The formation of grain boundaries with high angles.

These reactions occur during the restoration process of recovery. After this process, the following can occur:

Dislocations as well as point defects being absorbed as a result of the migration of high angle grain boundaries.

A decrease in the overall grain boundary area.

These micro-structural changes occur during the restoration process of recrystallization and recovery. As a result of these micro-structural modifications, an ideal definition of recrystallization is derived:

Along with the micro-structural changes, the properties of the specimen also change correspondingly. Thus, deformation and annealing are important processing methods for producing desired properties of the material by controlling its microstructures.

Recrystallization mechanism

The start of recrystallization is referred to as nucleation and occurs when dislocations are rearranged so as to form low dislocation density sections that have a high angle grain boundary with great mobility and thus is capable of quick movement over the strained region or recovered matrix. Recrystallization has a low driving force and high grain boundary energies; as a result of these characteristics, thermal variations cannot explain regions surrounded by high angle grain boundaries that are free from defects upon annealing. Therefore, the formation of recrystallized grains does not occur during annealing but previously exists in the deformed state. Three methods can be used to describe nucleation and they are:

Movement of high angle boundaries that already exist before annealing: this happens when pre existing grain boundaries move into grains that are highly strained as illustrated in figure 16 this process requires a favourable energy balance between an increase in the overall grain boundary surface and a reduction in stored energy as a result of the removal defects triggered by the migration of the boundary.

Movement of sub boundaries (that is low angle boundaries): this model is based on the theory of polygonization where stored energy is reduced during annealing as a result of rearrangement and removal of defects. It occurs when sub grain boundaries besiege regions containing low dislocation densities. Upon formation of sub grains, with the help of sub grain boundary movement, they are able to grow at the expense their neighbouring grains. Dislocations are absorbed by migrating sub boundaries and because of this, their mobility, orientation differences and energies are increased until their transformation into high angle boundaries, thus illustrating nucleation.

Sub grains coalescence: this occurs when two neighbouring subgrains merge leading to their crystal lattices coinciding. It is regarded as a slow process but when compared to migration of sub grains is favoured for annealing at low temperatures. it is illustrated in figure 17.

In this method, stored energy is reduced leading sub boundaries disappearing, sub grains growing and increase in orientation differences between coalescence groups and their neighbouring sub grains. These lead to the formation of high angle boundaries which move at high speeds and cause the process of recrystallization nucleation.

It is vital to identify the fact that the total energy balance that takes the disappearance of sub boundaries into account with the increase and orientation difference is favourable (that is it leads to a reduction in total free energy). This mechanism is illustrated in figure 18.

The occurrence of these three models is relatively diverse and they will therefore occur under different conditions. The basic requirement for the occurrence of the movement of pre existing grain boundaries that is the existence of differences in large strain between neighbouring grains is well accepted by researchers. However, there is conflict as to when the mechanisms sub grain boundaries migration and the coalescence of sub grains occur. Researchers believed the coalescence of sub grain boundaries are linked with large dispersion of sub grain angles distribution, relatively moderate strain, and reasonably low annealing temperatures. Whereas the mechanism of sub grain migration is linked with high annealing temperatures, strains that are relatively high and large dispersion in the distribution in sub grain size.

Growth of recrystallized regions

The basic mechanism causing recrystallization and grain growth is the migration of grain boundaries with high angles. However their driving force is what differentiates them from each other. The energy of the high angled grain boundaries is the main driving force for grain growth whether it being abnormal or normal growth. Whereas that for recrystallization is the energy stored during straining appeared as crystalline defects. In defect free regions that are encircled by boundaries with high angle, recrystallization progresses by enlargement of this nucleus over the non recrystallized medium. Grain

growth and recrystallization's migrating high angle boundary curvature signal is another important factor that differentiates the two.

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EXPERIMENTAL PROCEDURE

Equipment

Three samples of YTZP, One made from 3mol Y₂O₃/ZrO₂ powder and the other two made from the same powder but by two other manufacturers.

Focused Ion Beam (FIB): is a technique used by material scientists in the analysis of a materials microstructure. the samples obtained can either be analysed directly using the FIB or transferred and viewed under a TEM or

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SEM. This process is similar to that of an SEM however ions (particularly gallium ions) rather than electrons.

Figures 19a and b show how the FIB operates. The surface of the sample being tested is subjected to primary gallium ion beams. This spits a small part of the material, leading to the formation of either secondary ions (either positive or negative) or neutral atoms on the surface. Secondary electrons (e) are also produced from the gallium beam. This is collected as well as the signal from the split ions to form an image which is either analyzed using the FIB itself, SEM or TEM.

<http://www.fibics.com>

[com/fib/tutorials/introduction-focused-ion-beam-systems/4/](http://www.fibics.com/fib/tutorials/introduction-focused-ion-beam-systems/4/) images is from here.

GNU image manipulation program (GIMP): is an image editing software used to measure grain sizes.

Vickers indentation: is used to form indentations which are viewed and studied under an optical microscope. A square imprint is formed from the Vickers indenter, where the two diagonal lengths are measured. In this project, the Vickers indentation is used to form plastic deformation. The surface defects such as scratches and unevenness need to be controlled, hence the reason for polishing.

High temperature furnace: used for sintering and annealing.

TEM

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Cold isostatic pressing

Sample preparation

Three mol% Y₂O₃/ZrO₂ solid solution powder was used in this experiment. The powder was pressed into a disc at 200MPa, then sintered at 1450°C for 2 hours. Two discs were produced, each pressed with loads 7.5 tons and 5 tons respectively. The load was reduced to 5 in order to reduce the chance of lamination, as it occurred with the 7.5.

Measurements and dimensions

Sample 1: broken

Sample 2

Weight

25.7711g

19.6836g

Diameter

39.23

39.11

Height (thickness)

7.1

6.51

Dimensions after sintering <