Local Microstructure Parameters in Polycrystalline Materials and Design of Interfacial Microstructure of Joints†

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Abstract

Concepts of microstructures from the viewpoints of the statistic and the spatial distributions are explained concentrating on the local distribution or the spatial distribution of grain orientation. Then EBSP analysis of the microstructures of a FSW joint and a recrystallized sheet of Al alloy are explained as examples of the static and the dynamic evaluation of microstructures. The final topic is the design and control of interfacial microstructures of joints regarding the phase reaction at the interface between ceramics and metals. A research topic in nano-materials will be briefly mentioned.

KEY WORDS: (local microstructure parameter) (EBSP) (FSW) (grain boundary migration) (diffusion bonding) (nanocrystalline material)

1. Introduction

Joining and welding processes create unique microstructures in the joint region utilizing thermal, chemical, mechanical energies that work in a localized region. Therefore we can design microstructures in the weld region or the interfaces of dissimilar joints, which are different from those in the case of industrial mass productions such as casting, rolling and so on. In addition, differences in chemical potentials of constituents bring about the phase reactions at the interface of dissimilar joints, even for the case of a homogeneous temperature field. The present paper briefly describes one of our strategies for interface engineering.

2. Microstructure parameters and their spatial distributions

Microstructures of polycrystalline materials are described by important parameters such as (1) phases, (2)shape and size of grains, (3)crystallographic orientation of grains and (4)grain boundary structure and character. In addition, these parameters should be recognized in terms of both statistical and spatial distributions.

Figure 1 depicts the concept of local microstructure parameters along with the statistical one called *"macro-texture"* [1]. In particular the spatial distributions of both grain orientation (*microtexture*) and grain boundary character [2] (*meso-texture*) have been attracting much attention since texture formation is affected by these distributions.

Likewise our fingerprints and genome information are identical, every specimen has unique microtexture and other parameters and is different from each other. Moreover such microstructure information can be decoded in the post welding treatment, suggesting that the joining

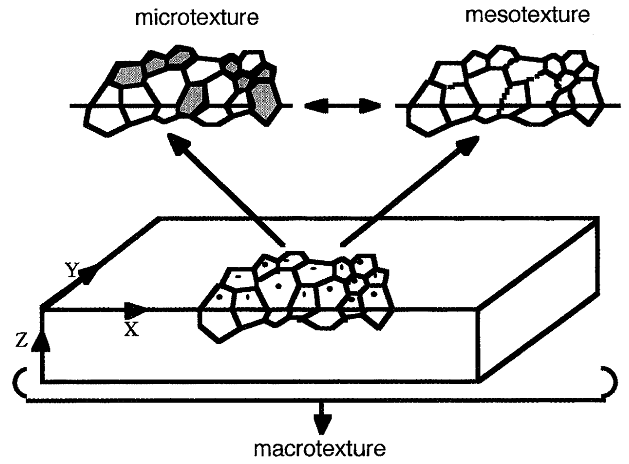

Fig. 1 Distributions of microstructure parameters in polycrystalline materials [1].

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process codes the important information into a localized area. Actually we have not succeeded in deciphering such cryptograms so far, but are now struggling to solve this problem.

3. Static and dynamic microstructure parameters

Taking into account the evolution of microstructure, there are two categories of microstructure characterized by (1) static and (2) dynamic parameters. Usually we use the former parameters for the characterization of given materials, but as mentioned in the previous section we need to know the microstructure that is evolving even just right now. The *Electron Back-Scatter Pattern* (EBSP) method can give us useful information [1].

Figure 2 represents an example of EBSP analysis dealing with the static microstructure parameters in the FSW joint of A5083/6061 Aluminum alloys. The microtexture, represented by unique colors corresponding to the normal direction of each grain, consists of several components of orientation close to red colored <001>, blue colored <111> and others. Each grain belonging to the same orientation group tends to form "clusters", and some clusters seem to be lying along the direction of plastic flow caused by the rotation of the pin. These spatial distributions are different depending on the area in the joint region observed, suggesting that we are facing some new metallurgical phenomena of the simultaneous events of complicated metal flow and dynamic recrystallization process during FSW. In other words, we have obtained an advanced method of microstructure control that enables us to fabricate peculiar microstructures in limited regions by utilizing FSW or friction stirring in the surface region, and now our group is trying to develop and refine these techniques.

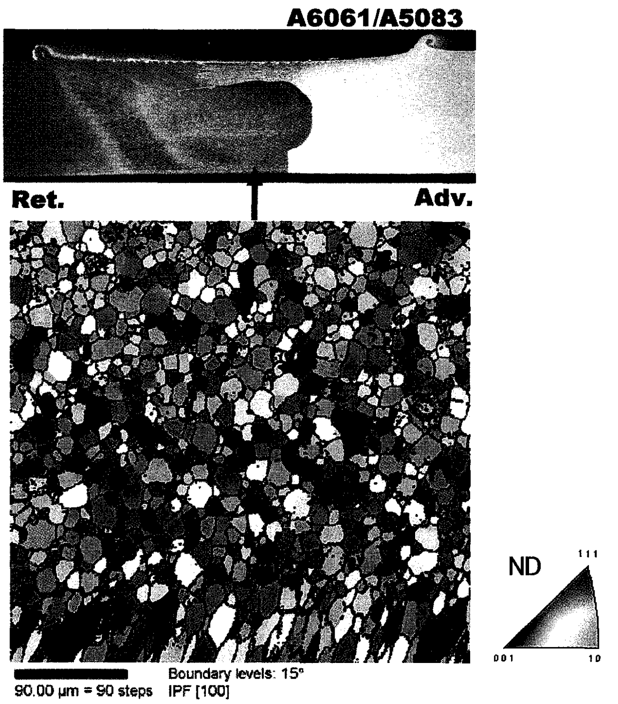

Fig. 2 OIM analysis of the stir zone of the FSW joint of A6061/A5083 Al alloys.

The dynamic information on microstructure can be obtained by utilizing *double etching technique.* **Figure 3** represents a back-scattered electron image (BEI) showing an example of such an evaluation performed for the Al-4Mg alloy sheet recrystallized at773K after cold rolling [3]. In this figure, both the original position and the present

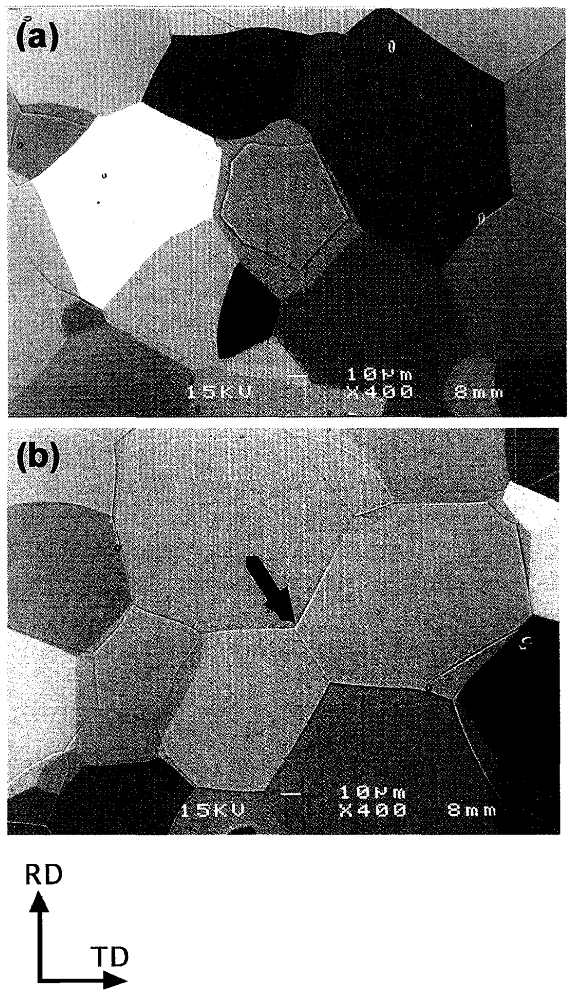

Fig. 3 BEIs of Al-4mass%Mg alloy sheet recrystallized at 773K.
(a) An example of moving boundaries and active triple junctions.
(b) An example of stable boundaries and triple junction indicated by an arrow.

position of grain boundaries and triple junctions are visible. Since the slight etching of the specimen after the recrystallization treatment made a shallow groove of each boundary, the grooved line represents the former position. The present position of boundaries is recognized by the difference of darkness in the BEI, since the contrast of the BEI is sensitive to the grain orientation. Many boundaries and triple junctions are active and migrating, but not all. This result means that the evolution of microstructure proceeds not in a homogeneous manner, and it strongly depends on local microstructures such as grain boundary characters around each triple junction. This technique enables us to know the active and passive area in the given specimen. The EBSP data will become more effective for the prediction of microstructure change by coupling the computer simulation of grain growth [4]. We do need precise information of each grain boundary in terms of boundary energy, boundary mobility, segregation, precipitation and so on, that are functions of the atomic structures of grain boundaries. This dynamic analysis of microstructure is also applicable for the evaluation of the interfacial microstructure of joints.

4. Design of interfacial microstructures of joints

Interfacial microstructure should be designed by taking into account the role of macro-, micro- and meso-textures as described previously. On the other hand, the reactive interfaces and joints of nanostructured materials are also important targets to be investigated by our group. Reactive interfaces between dissimilar materials such as ceramics and metallic materials often yield brittle reaction products that reduce the strength of joints. Therefore the phase reaction should be controlled, and we have been utilizing both thermodynamics and diffusion to understand and to describe the phenomena that are proceeding at the interfaces. For example, the joint of Si_3N_4/Ti yields Ti_5Si_3 which is the main reason for the low strength of this joint, but the dissolution of nitrogen into Ti was found to suppress the phase reaction and finally we have succeeded in improving the fracture strength by a factor of three [5]. The mechanism of the suppression is explained in terms of a chemical potential diagram. The utilization of chemical potential diagrams and other thermodynamic parameters makes the design of interface more precise and productive.

Nano-materials attract many researchers and companies who have been chasing novel properties in

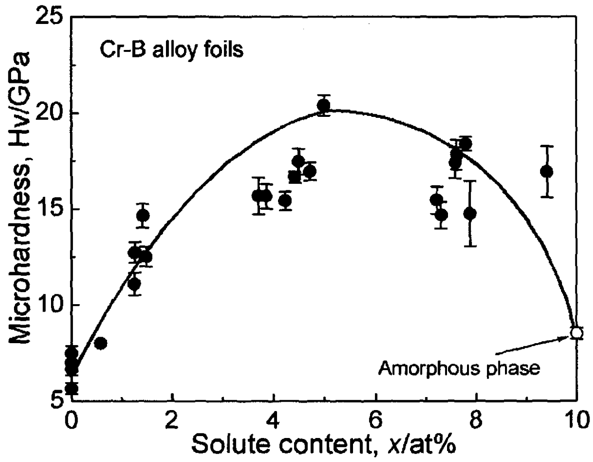

Fig. 4 Changes of microhardness as a function of solute content in Cr-B alloy foils deposited by RF sputtering [6].

nano-size. Joining of these nano-materials is a key technology to fabricate micro- and nano-machines that will be a final goal of nano-engineering. For this purpose, we have been firstly trying to reveal the mechanical properties of nanocrystaline foils prepared by the RF sputtering method. Nanocrystalline foils of Cr-B and Cr-Ni alloys have been revealed to possess two distinct features such as the existence of an amorphous phase in the grain boundary region and a critical grain size where the material loses the Hall-Petch relationship between microhardness and grain size [6, 7]. **Figure 4** shows the changes of microhardness with increasing amount of B in Cr-B alloy foils prepared by the RF sputtering method. The decrease of hardness is mainly caused by the emergence of a grain boundary amorphous phase in the solute content region higher than 5 at%.

Now we are moving on the second step, such as the mechanical behavior of nano-materials at elevated temperature followed by the final goal of the design and control of the interfaces in nano-joining.

5. Summary

Microstructure in polycrystalline materials contains much information such as statistical distributions, local or spatial distributions, static and dynamic microstructure parameters. Joining technology needs to optimize these parameters for given materials in order to fabricate sound joints. Some new techniques like EBSP analysis and grain growth simulation are effective for the evaluation of interfacial microstructures, and basic research on interfacial properties will be required.

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