The International Journal Of Engineering And Science (IJES)
||Volume||2 ||Issue|| 6 ||Pages|| 12-18||2013||
ISSN(e): 2319 – 1813 ISSN(p): 2319 – 1805

Characterization of Cyclically Deformed Persistence Slip Bands and Ladder like PSB's in Copper Grain Structure

Aezeden Mohamed, Wei Liu and M. N. Bassim
Faculty of Engineering, University Manitoba, Winnipeg, Canada, R3T 5V6

ABSTRACT

A study of microstructural characterization of copper polycrystals during strain control fatigue has been studied for three decades. However, the reported results are quite controversial in regard to the plateau behavior, which has been well established for single copper crystals. The absence of complete and reliable results on the effects of the copper grain size when polycrystals are subjected to strain control fatigue testing was the primary motivation for the present study, with the goal being to address the cause of a clear answer in the existing literature. Saturation stresses measured in strain control fatigue tests were plotted as a function of corresponding plastic strain amplitudes to obtain a cyclic stress strain curve (CSSC) for three different grain sizes. After testing, cycled specimens were sectioned and cut longitudinally in the gauge length section of the specimens to produce samples for etching and microscopic investigation. Scanning electron microscopy (SEM) was conducted on the etched samples to determine the presence of persistent slip bands (PSBs) in each grain size.

KEYWORDS - *Cyclic, curve, fatigue, grain, persistence, plateau, saturation, slip, strain, stress.*

Date of Submission: Date, 12 June 13 ⟷ Date of Publication: Date 25 June 13

I. INTRODUCTION

For three decades the research on fatigue has examined the fracture behavior, i.e., crack propagation and fracture mechanics. This research has been extremely valuable for failure analysis and in the fracture control of structures. However, much of this work has been carried out from an engineering point of view and less attention has been given to the basic mechanisms of fatigue. It is recognized that without a fundamental understanding of such basic processes, designing structure with high her safety would be difficult. Klesnil [1] reported that the fatigue process is completely dominated by cyclic plastic deformation, which causes irreversible changes in the material microstructure. In general, the fatigue process can be divided into three stages: fatigue hardening and /or softening, micro crack nucleation, and crack propagation. The final stage has received the most attention and can be considered to be well established. On the other hand, the first two stages, where most of the fatigue problems occur in many types of structure, have received less attention and there are still a lot of unsolved problems.

Since the 1970's there have been many studies on the cyclic stress strain behavior of pure metals and alloys. Most of these have used single crystals because it is helpful and easy to understand basic fatigue behavior of a single crystal before studying polycrystalline materials.

Throughout those investigations, such as strain control fatigue test and corresponding mechanism, has been achieved. Over the past two decades the cyclic deformation behavior of polycrystalline materials has also been studied extensively. However, investigations have given rise to a considerable scatter in the reported results. In order to provide some clarification in our understanding of the behavior of material under cyclic deformation, the present study investigated the microstructural characterization of copper with different grain sizes subjected to stain control fatigue.

II. EXPERIMENTAL PROCEDURES

The material used for the tests was pure commercial copper (99.94%Cu, < 0.01%Al, < 0.005%Si).
As received copper rod with a diameter of 12.7mm was machined into fatigue specimens with a total length of 115mm and a gauge length of 20mm with a 7.4mm diameter.

Three heat treatment schemes were applied to obtain three sets of specimens with different grain sizes of 22μm, 35μm, and 100μm as shown in Figures 1 (a), 1 (b) and 1 (c), respectively. A tube furnace with an argon protection atmosphere was used for the heat treatments in order to minimize surface oxidization. Pre-strain was conducted using a tensile testing machine to produce a specimen dimension of 120mm and a gauge length of 25mm with a 6.5mm diameter. All specimens were divided into three groups. The first group was heat treated at 566°C for 0.5 hours to produce a 22μm-grain size, the second group was heat-treated at 650°C for 2 hours to produce a 35μm grain size, and the third group was heat treated at 866°C for 1.5 hours to produce a grain size of 100μm. Only the first and second groups (22μm and 35μm specimens) were pre-strained by 30 % plastic elongation and furnace-cooled to eliminate internal stresses and enable them to be pre-strained by a relatively large plastic elongation. The plastic pre-strain was carried out on an INSTRON screw-driven tensile testing machine, which was connected to an IBM XT personal computer for sampling and processing of test data.

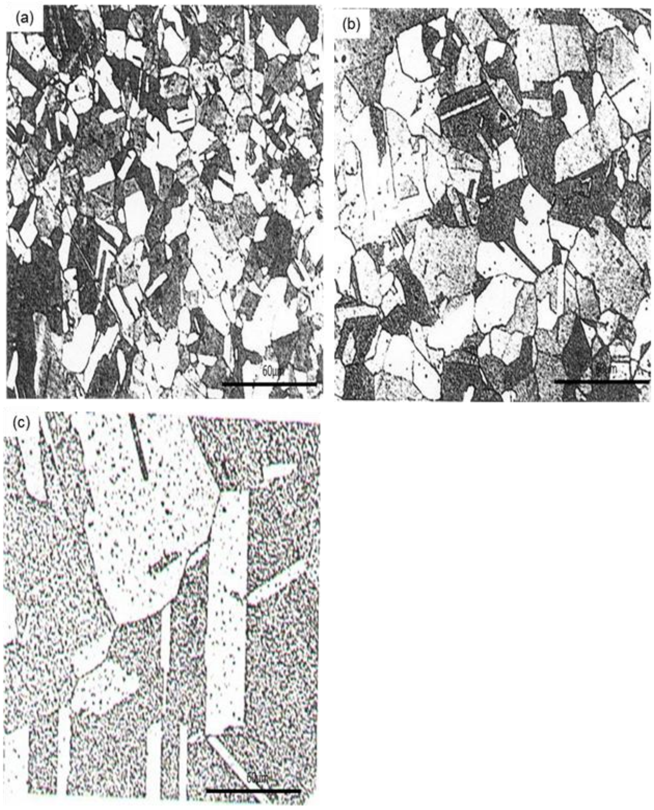

Figure 1: Optical micrograph of copper with grain sizes of (a) 22µm, (b) 35µm, (c) 100μm.

III. RESULTS

3.1 Cyclic stress strain curves

The cyclic stress strain curves (CSS) for grain sizes of 22μm, 35μm, and 100μm were compared in Figure 2. In addition, the current testing methodology followed similar methodology to Liu [2] whose results are also provided for comparison.

It can be seen that all CSS curves exhibit three regions of cyclic hardening and a quasi-plateau region. However, a grain size effect is also noticed: 1) except at very low plastic strain range, a smaller grained polycrystalline copper clearly exhibits a higher saturation stress than that for a coarser grained polycrystalline copper, 2) a fine grained copper exhibits a steeper quasi plateau than a coarser grained copper 3) the quasi plateau region for a fine grained copper starts earlier and ends later, compared to a coarser grained copper.

The extension of the quasi plateau region is given in terms of strain amplitude from the onset of the plateau to its end. The fatigue limit, I, is defined as the saturation stress and the corresponding plastic strain amplitude below which the persistent slip bands (PSB) is absent; because no PSB formed just below the onset of a quasi-plateau (I).

The slope of the quasi plateau is defined as the change of saturation stress (MPa) with the plastic strain amplitudes ($\Delta\varepsilon_{pl.}/2$). From Figure 2 shows that the slope of the quasi plateau is steeper for a finer grained polycrystal than for a coarser one.

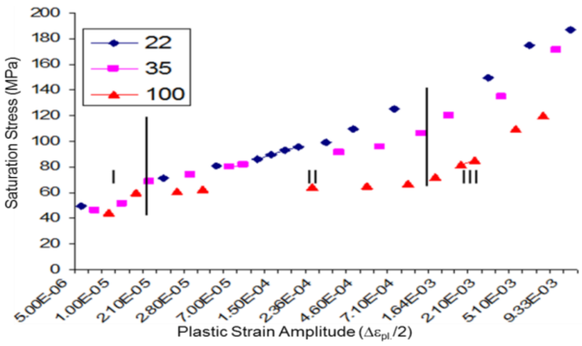

Figure 2: CSS Curves of Copper of with grain sizes of 22μm, 35μm, and 100μm.

3.2 Microstructural Characterization

The SEM investigation showed: 1) In region (I), no PSB's were observed, and 2) In region (II), PSB's started to form at the lower ends of the quasi plateaus. In general, the PSB population increases with the applied plastic strain amplitude ($\Delta\varepsilon_{pl.}/2$). However, the increase in population for smaller grained copper is not as fast as that for a coarser grained copper. At the end of the quasi plateau, less than 8% of the grains contain PSB's in 22μm-grained copper (Figure 3 (a)), around 12% of grains contain PSB's in 35μm grained copper (Figure 3 (b)), while for 100μm grained material, more than 30% of grains contain PSB's (Figure 3 (c)). An important item of information from the SEM investigation is that the PSB's in fine grain copper distribute more randomly compared to those in 100μm grained copper where most of the grains containing PSB's were fully filled with them. In 22μm grain copper most of the grains contain only one or two cluster of PSB's.

IV. DISCUSSIONS

4.1 Grained size Effect on CSS Response

Microstructural characterization of copper of three grains sizes reveals three regions of cyclic hardening. In region (I), and region (III), which correspond to low and high plastic strain regions, respectively, saturation stress increases markedly with increasing plastic plateau.

The occurrence of a quasi-plateau region in the CSS curve of polycrystalline copper was reported by Figueroa and Mughrabi [3, 4]. Liu [2] claimed a quasi plateau region in the CSS curve of 42μm grained structure copper. Liu's results are comparable to that reported in this paper. This is likely due to the use of the same testing conditions. Mughrabi [4] does not claim quasi plateau in his investigation on 25μm grained structure copper, and indicated three regions of cyclic hardening and a clear quasi plateau exist in the intermediate region.

A recent study by Mayer and Laird [5] indicates that testing frequency significantly influences the cyclic behavior of copper polycrystals. They found that low cyclic frequency stimulates PSB transformation from loop patches. In other word, low cyclic frequency promotes localization of deformation. Since it is well known that the formation of PSB influences cyclic response by reducing the slope of cyclic hardening and both the present study and above-mentioned studies were performed under low frequency, then it is not surprising that a quasi-plateau exists. These studies are congruent in their outcomes. Moreover, work by Figueroa [3] that resulted in a true plateau was also carried out at low testing frequencies.

After reviewing the CSS curves of copper polycrystals published in the literature, the test conditions can be classified into two broad categories of low constant strain rate (<E-02/second) and high constant frequency (>10Hz).

The plateau like behavior was more pronounced under low constant strain rate but tests were carried out at constant high frequency showed less plateau behavior or even no plateau behavior as reported by Mayer, Basinski and Mulliner [5-7].

In the current work with 22μm, 35μm, and 100μm grain sizes of copper polycrystals were used as shown in Figures 1 (a), 1 (b), and 1 (c). In the current work, as can be seen from Figure 2 (a), a fine-grained copper clearly exhibits a higher saturation stress ($\Delta\sigma_s/2$) than a coarse grained copper except at very low ($\Delta\varepsilon_{pl.}/2$). The extension of the quasi plateau reveals a grain size effect as well: the coarser the grain size, the longer the extension of the plateau. In addition, the quasi plateau region shows a different slope for each grain size.It is well known that the effect of grain size on monotonic deformation is described through models such as the Hall-Petch effect. This effect is due to the influence of grain size in limiting slip length, dislocation density and dislocation pile-ups, as reported by Boutin and Thompson [8, 9].

In general, flow stress is found to be proportional to the square root of dislocation density and that dislocation density is inversely proportional to slip length. At low strain the dislocation slip length approaches the dimension of the grain. Therefore, as grain size decreases, the flow stress increases. As was demonstrated for the copper in this study, persistent slip bands nucleated at the grain boundary and extended toward the opposite grain boundary: most slip bands reached the opposite side. For these reasons, it follows that a small-grained copper reveals a higher ($\Delta\sigma_s/2$) value. At higher values of ($\Delta\varepsilon_{pl.}/2$), where dislocation structures, the slip length approaches the cell dimension. Boutin [8] discovered that the grain size effect in monotonically deformed copper diminishes or even reverses at high strains.

Recent results by several investigators [7-9] agree on the effect of grain size in the cyclic response of copper polycrystals. It is known that a relatively open vein structure is formed at low ($\Delta\varepsilon_{pl}/2$), so slip length may be a function of grain diameter, which means grain size may be expected to influence saturation behavior. However, at very high ($\Delta\varepsilon_{pl}/2$) amplitudes a well-defined cell structures forms. Since grain size does not show the influence of cell size, grain size would not be expected to influence saturation behavior at very high amplitudes. This behavior in cyclically deformed copper was reported by Hirth [10]. The results of the present study generally reflect the typical behavior, namely, higher ($\Delta\sigma_s/2$) for smaller grain size. However, at very low ($\Delta\varepsilon_{pl}/2$), grain size effect on ($\Delta\sigma_s/2$) is not well defined and may be attributed to mechanical error or other explanations, which are left open for future study. It should be mentioned that the present study was not performed up to very high ($\Delta\varepsilon_{pl}/2$) amplitude, where cell structure is well developed. However, it can be clearly seen that the ends of the CSS curves are approaching each other (Figure 2), and might intercept at higher ($\Delta\varepsilon_{pl}/2$), where cell structure is well developed.

As demonstrated in CSS curves (Figure 2), the extension of the quasi plateaus shows a grain size effect. The quasi plateau started earlier and ended later for a coarser grain size compared to a finer grain size. The onset of a quasi plateau indicates the formation of PSB's by the breaking down of vein structure, and secondary slip bands begin development as a consequence of further application of ($\Delta\varepsilon_{pl}/2$). A study of these behaviours was reported by Hirth [10].

The hard region is caused by multiple slip produced by plastic incompatibilities from an adjacent grain and enhances homogenization of deformation until the whole grain becomes hard. The soft region has a single slip character at low intermediate strain amplitudes, and is associated with localization of deformation by formation of PSB's in cyclic deformation.

A study by Liu [2] of microstructural characterization using transmission electron microscopy (TEM) on cyclic saturated polycrystalline copper with a 42μm grain size, conducted in region I, noted cylindrical loop patch structures in single crystals and coarse grained polycrystals.

Moreover, the irregular loop patch and cellular loop patch, which were rarely reported to occur in coarse grained copper and single crystals, were observed in region (I). In region (II), the dislocation structure formed veins and PSB's structures were also present in this region, but with much lower fractions than that found in the coarse grain size. Both of the above mentioned studies indicate that the smaller the grain size, the more pronounced multiple slip behavior, which leads to more homogenous deformation.

The results of the present study can be explained in similar terms as Liu's [2] study. Since a coarse grain can be interact with a bigger volume fraction of a soft region oriented for single slip than a smaller grain, more PSB's are than for a smaller grain. On the other hand, a small grain has a bigger volume fraction of hard region oriented for multiple slip, and develops more homogenous deformation. The constraint effect is also more significant because of the compatibility requirement, and thus the applied deformation can be distributed among the whole grains more effectively. Therefore, the formation of PSB, which indicates the start of the quasi plateau, might need larger ($\Delta\varepsilon_{pl}/2$) and ($\Delta\sigma_s/2$) to be applied for small grained polycrystals. For coarse-grained polycrystals, the constraint effect from the adjacent grain is less significant because the volume fraction of the hard region is small compared to that for a smaller grain and conditions are more favorable for localization of deformation rather than homogeneity of deformation. For a small grain, the effect is opposite. The compatibility requirement is more pronounced and the localization of deformation can be suppressed more easily because of a significant constraint effect resulting from the higher density of grain boundaries; multiple slip behavior in promoted.

4.2 Grain Size Effect on PSB Formation

PSB's formed during the cyclic deformation of the polycrystalline copper show short and straight thin lines, which were broken down from the surrounding matrix. The lines nucleated at a grain boundary without directly crossing it. Occasionally, it may be observed that PSB's in two adjacent grains intersect each other but proceed in different directions. These common features, which were also observed by Liu and Mayer [2, 5] indicate that grain boundaries play an important role in the formation of PSB's.

4.2.1 PSB's in Fine Grain Size Copper

The investigation on smaller grain sizes of 22μm and 35μm as shown in Figures 3 (a) and 3 (b), respectively, shows that the PSB population increases with increasing $\Delta\varepsilon_{pl}/2$ amplitude along the quasi plateau region. However, even at the ends of quasi plateau regions fewer than 10% of the grains contain this ladder like structure. The PSB's formed in small-grained polycrystals are randomly distributed in the bulk of the polycrystals. The results obtained from the present study support Bassim's investigation on 42μm grain size [2].

Liu [2] concluded that PSB's are not the governing microstructure in fine grained copper since only a few grains can orient with the primary slip system and are favorable to build up the ladder like PSB's; the major contribution to the saturation stress is from the deformation of the bulk matrix. When the vein structure of the matrix breaks down and forms PSB's in some grains, it reduces the stress sustained by these grains. However, the decrease of the stress on the grains leads to the redistribution of stresses among all grains and results in a stress rise in the neighboring grains. Therefore, because of the high constraint inherent with a small grain size, "location of deformation is not easy to occur" and "matrix received most of applying ($\Delta\varepsilon_{pl}/2$)."

To ensure a high-quality product, diagrams and lettering MUST be either computer-drafted or drawn using India ink. Figure captions appear below the figure, are flush left, and are in lower case letters. When referring to a figure in the body of the text, the abbreviation "Fig." is used. Figures should be numbered in the order they appear in the text. Table captions appear centered above the table in upper and lower case letters. When referring to a table in the text, no abbreviation is used and "Table" is capitalized.

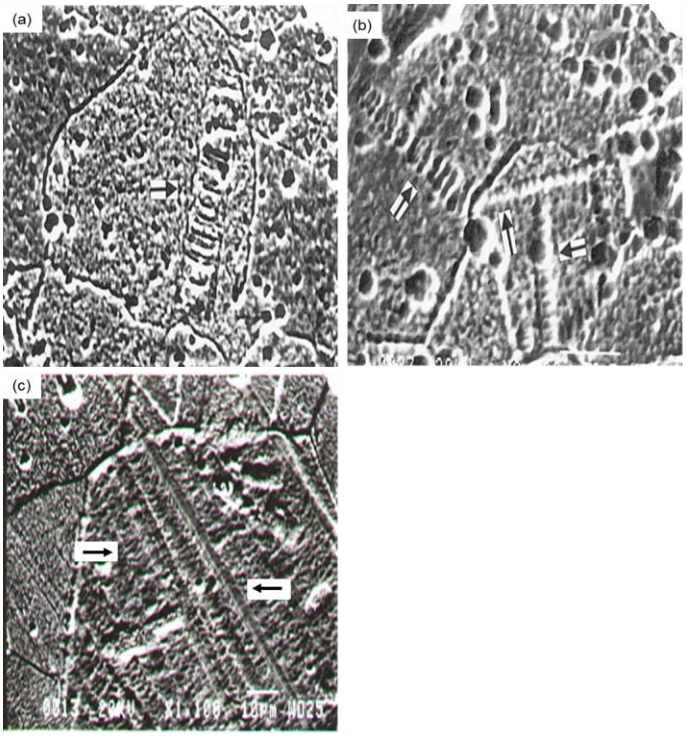

Figure 3: SEM micrographs showing (a) less PSB's for 22μm grain size, (b) more PSB's for 35μm grain size, and (c) fully filled with PSBs for 100μm grain size. All samples were taken from the intermediate region (II).

Liu [2] asserted that the two phase model, which regards the PSB structure as the soft phase that determines the saturation stress on the bulk of material, is not applicable in polycrystals. He made an assumption that the PSB's can be considered to mix up (aggregate) with the matrix in the manner of a composite, so that the saturation stress on the bulk of the polycrystals may be assumed to obey a mixture law.

The CSS curves for fine grained 22μm and 35μm copper in this study shown in Figure 2, show quasi plateau regions. The microstructure shows that the quantity of PSB's is very small even at the upper end of the quasi plateaus, so the saturation stress is mainly contributed by a combination of matrix structure and PBS structure. Moreover, the quasi plateau region for 22μm grained copper is steeper than that for 35μm grained copper as exhibited in Figure 2. The volume fraction of PSB's can explain this phenomenon. It is larger for 35μm grained copper (Figure 3 (b)) than that for 22μm grained copper (Figure 3 (a)).

4.2.2 PSB's in Coarse Grained Size Copper

A very flat quasi plateau was observed in the CSS curve of 100μm grained copper as shown in Figure 2. The SEM microstructure shows that the PSB population increases linearly with increasing $\Delta\varepsilon_{pl}/2$ along the quasi plateau and up to approximately 30% of the grains contain ladder like PSB's at the end of the quasi plateau. Moreover, some of the grains are almost fully filled with PSB structures as shown in Figure 3 (c).

True plateau behavior in the CSS curve is well known for copper single crystals as reported by Mayer [5]. The onset of the plateau is marked by the formation of PSB's; the PSB density increases linearly as the strain amplitude increases and at the end of the cycled deformation depended on the stress sustained by the PSB's, which means that PSBs are responsible for and dominate the plateau behavior.

True plateau behavior of polycrystalline copper was reported by Kuokkala et al [11], who found a plateau region in his study of 150μm grain size polycrystals, More than 60% of grains contained PSB's at plastic strain amplitudes near to the upper end of the plateau. They suggested that the dislocation structure in coarse-grained polycrystals was qualitatively similar to that of single crystals. The PSB pattern shows a similar tendency for individual grains to avoid PSB on more than one slip system, which indicates the plastic strain is carried mainly by PSB's with single slip.

The explanation for the occurrence of a plateau region was proposed by Mughrabi [4], who studied copper bicrystal. Based on TEM investigation, Mughrabi [4] found that the grain boundary acted as an obstacle to the motion of dislocations and at the end of the plateau region a secondary slip system was activated. However, the population of the PSB's increases linearly and rapidly along the plateau. Also, the phenomenon of activation of secondary slip systems at the end of the plateau was also observed in single crystals. Therefore, we can safely state that as long as the PSB's are predominant, the occurrence of a plateau should be possible in the CSS curve.

The present study on 100μm grained copper shows an almost horizontal quasi plateau, where the ($\Delta\sigma_s/2$) increases very slowly with increasing ($\Delta\varepsilon_{pl}/2$), compared to the less horizontal plateau for 22μm and 35μm grained materials as shown in Figure 2. The composite (matrix and PSB's) provides a reasonable explanation for the behavior of fine grain copper in the current study, where the combination of matrix structure and PSB's control the saturation stress. However, for coarse-grained copper, the contribution of PSB's to the saturation stress is larger, and plays a more important role on cyclic response.

It may be comfortably stated, based on the present study that the smaller the grain size, the higher the fatigue limit. This is consistent with observation that the coarse grained material is more likely to be deformed by single slip whereas multiple slip plays a more important role in the deformation of fine-grained material.

V. CONCLUSION

The CSS curves of different grain sizes in copper exhibit three regions of cyclic hardening, in which a quasi-plateau region occurs where the saturation stress increases less significantly with increasing plastic strain amplitude than for regions (I) and (II).

In general, a finer grained copper shows a higher saturation stress. The difference in saturation stress is primarily caused by higher back stresses in the finer grained material. However, this trend is undefined in the very low plastic strain region. In region (I) the CSS curves are approaching each other, thus indicating that grain size might lose its effect at higher plastic strains.

The quasi plateau regions show a grain size effect in that: (a) the larger the grain size, the earlier the beginning and the latter the ending of the quasi plateau in terms of the plastic strain, and (b) the smaller the grain size, the steeper the quasi plateau.

SEM investigation shows that grain size influences the slip character of the material. PSB's occur at lower plastic strain amplitudes and the secondary slip bands occur at higher plastic strain amplitudes in a coarse grained copper.

The fact that a PSB occurs at higher plastic strain amplitude and saturation stress indicates a greater fatigue limit for fine-grained copper polycrystals.

REFERENCES

[1] M. Klesnil and P, Luckas, Effect of stress cycle asymmetry on fatigue crack growth, *Materials Science Engineering, Vol. 9*, 1972, pp. 231-240.

[2] C. D. Liu, D. X. You and M. N. Bassim, Cyclic strain hardening in polycrystalline copper, *Acta Metall. 42*(5), 1994, pp. 1631-1635.

[3] T.C. Figueroa, S.P. Bhat, R. De La Veaux, S. Murzenski and C. Laird, The cyclic stress-strain response of copper at low strains—i. constant amplitude testing, *Acta Metallurgica, Vol. 29*(10), 1981, pp. 1667–1678.

[4] H. Mughrabi and R. Wang, *Mechanisms and Microstructure, Proc. 2nd Riso Int. Symp. On Metallurgy and Materials Science*, 1980, pp. 90-91.

[5] H. Mayer and C. Laird, Frequency effects on cyclic plastic strain of polycrystalline copper under variable loading, *Material Science and Engineering A 187*(2), 1994, pp**.** 137-145.

[6] Z. S. Basinski and S. J. Bansinski, Copper single crystal PSB morphology between 4.2 and 350 K, *Acta Metallurgica, Vol. 37(12), 1989,* pp. 3263–3273.

[7] H. Mulliner, B. Weiss, R. Stickler, P. Lukas and L. Kunz, On fatigue and fatigue thresholds, *Fatigue 84. Proc. Second Int. Conf.,* ***1****,* 1984, pp. 423-424.

[8] J. Boutin, N. Marchand, J. P. Bailon and J. I. Dickson, An intermediate plateau in the cyclic stress-strain curve of α brass. *Materials Science and Engineering, vol. 67*(2), 1984, pp. L23-L27.

[9] A. W. Thompson, M. I. Baskes, and W. F. Flanagan, The dependence of polycrystal work hardening on grain size, *Acta Metall., 21*, 1973, pp. 1017–1028.

[10] J. P. Hirth, The influence of grain boundaries on mechanical properties, *Metallurgical Transactions, Vol. 3(12)*2, 1972, pp. 3047-3067.

[11] V.-T. Kuokkala, T. Lepistö, and P. Kettunen, Random strain cycling of large-grained polycrystalline copper, *Scripta metal, Vol. 16(10)*, 1982, pp. 1149–1152.