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Microstructural and hardness gradients in Cu processed by high pressure surface rolling

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Abstract. The surface of an annealed Cu plate was processed by a high pressure surface rolling (HPSR) process. It is found that the deformed surface layer in the Cu plate after HPSR can be as thick as 2 mm and is characterized by a gradient microstructure, with grain sizes varying from the nanoscale in the topmost surface to the microscale in the bulk. The hardness varies from 1.37 GPa at the topmost surface to about 0.6 GPa in the coarse-grained matrix. The results of the investigation demonstrate that the HPSR process shows good potential for the generation of thick gradient microstructures on the surface of bulk metallic materials.

1. Introduction

In the past two decades, various surface deformation techniques, such as surface mechanical attrition treatment (SMAT) [1, 2], surface mechanical grinding treatment (SMGT) [3, 4] and high energy shot-peening [5], have been developed to generate surface gradient layers in metallic materials. Using these techniques, a gradient microstructure with a variation in grain size along the depth direction is typically formed in the surface region of initially coarse-grained material [6]. Such a surface gradient microstructure significantly increases the hardness [2], and improves both wear resistance [7] and other surface properties [3], leading to an extended service lifetime [8]. A superior combination of strength and ductility can also be achieved by introducing a surface gradient nanostructure [9]. However, the thickness of the deformed surface layer with a gradient nanostructure is often quite limited, being a few hundred micrometers in most cases [4, 9]. To further explore the potential of gradient microstructured materials, techniques that produce thicker layers of gradient microstructure are highly desirable. In the present study, a technique called high pressure surface rolling (HPSR) is presented. To demonstrate this technique HPSR has been applied to a Cu plate and the resulting microstructure and hardness are examined and discussed.

2. Experimental

The material used in the present study was a commercial purity copper (99.95%) disk plate with a diameter of 60 mm and a thickness of 6 mm. The disk was fully recrystallized with a grain size of few hundred micrometers after annealing at 500 ºC for 2 h. A newly developed technique, namely the high pressure surface rolling (HPSR) process, was used to deform the copper disk plate. The HPSR procedure is schematically illustrated in figure 1a. Three groups of cylinder rollers (GCr15, Ø8 mm ×

10 mm), containing six rollers in each group, are pressed into the sample surface under a load. Then the sample platform is rotated, leading to rotation of the rollers and rolling deformation of the sample surface. The repeated rolling of the rollers on the surface of the sample leads to highly localized plastic strain in the surface layer of the sample. As a result the grains are gradually refined and a depth dependent microstructure is generated. Detailed information about the HPSR process can be found in Ref. [10]. In this study, a load force of 20 kN and a rotation speed of 2 rpm were used. Each session of the process was interrupted after a 5 min treatment time and the sample then cooled in liquid nitrogen to reduce the temperature increase during HPSR. In total six sessions were applied to the plate to effectively refine the grains in the surface and increase the thickness of the nanostructured surface layer.

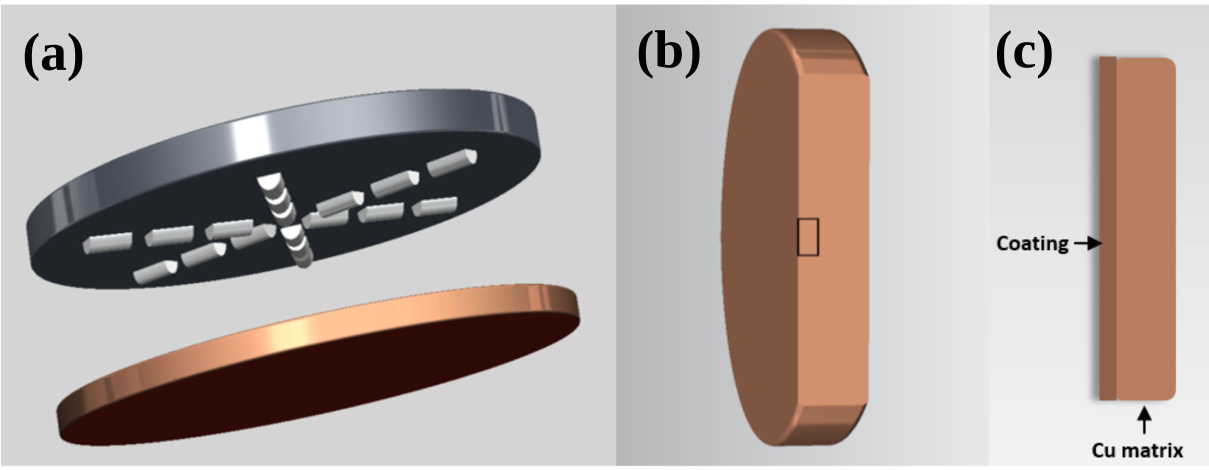

Figure 1. Schematic illustration of (a) the experimental set-up for high pressure surface rolling of a Cu plate; (b) the cross-section used for microstructural observations and hardness tests; (c) the coating of a protective Cu layer on the treated surface.

A specimen was cut from the rolled plate at a distance of 25 mm to the center of the plate (as indicated by the black box in figure 1b), and then a protective Cu layer was electrodeposited on the treated surface (as indicated by darker shading in figure 1c). The hardness variation along the normal direction from the surface was measured using a load of 20 g and a loading duration of 10 s. In the top 250 µm of the sample hardness indents were taken at intervals. The nearest indent to the top surface was taken at a depth of ~15 µm. At least 10 indents were measured at each depth. The cross-sectional microstructure of the HPSR sample was characterized by electron channeling contrast (ECC) and electron backscatter diffraction (EBSD) in a TESCAN MIRA 3 scanning electron microscope. Samples for EBSD were mechanically polished and then electropolished in a solution of phosphoric acid (25%), alcohol (25%) and deionized water (50%) at room temperature. To explore the deformed microstructure in different locations, a step size of 20 nm was used for depths less than 300 µm, and a large step size (100-200 nm) was used at larger depth. The variation in boundary spacing as a function of depth from the surface was also determined from the EBSD data. Values of boundary spacing were taken from line scans along the normal direction using a minimum misorientation of 1^{o} for identification of boundaries.

3. Results and discussion

The deformed microstructure after HPSR is shown in figure 2. It is clearly seen in the low magnification ECC image (figure 2a) that a deformed layer of about 2 mm in thickness is formed on the coarse grained substrate without a clear interface between the deformed layer and the matrix. A typical gradient structure along the depth from the surface is clearly observed although the detailed microstructure in the deformed surface is too fine to resolve by ECC at this magnification. Figure 2b shows the hardness variation as a function of depth from the surface. The plate shows a steep hardness gradient to a depth of 2000 μm from the surface. The average hardness is as high as 1.37±0.03 GPa in the topmost surface, which is significantly higher than that of the initial coarse-grained (CG) condition (0.60±0.02 GPa), as indicated by the horizontal dashed line. The hardness decreases gradually with increasing distance from surface, and approaches that of the un-deformed coarse grains at a depth of 2000 μm. A plateau in hardness values is observed at a depth of 300-600 μm.

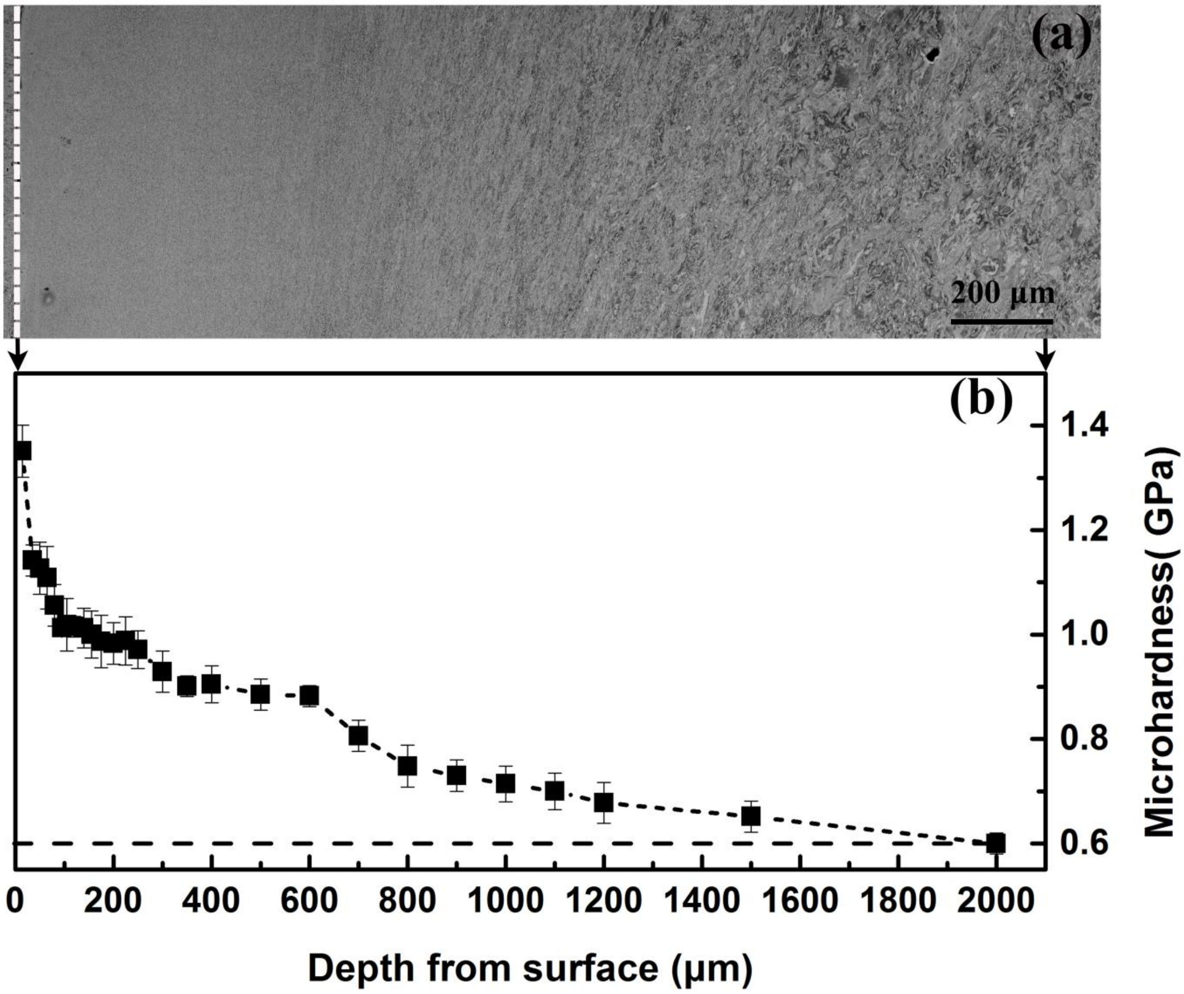

Figure 2. (a) ECC image in the longitudinal section of the HPSR Cu sample. The white dashed line indicates the position of the surface. (b) Vickers hardness as a function of depth from the surface. The horizontal dashed line indicates the initial hardness of 0.6 GPa. Note the correspondence between the image in (a) and (b) as marked by a pair of arrows.

To reveal the microstructure in the topmost surface layer of the HPSR processed Cu, high magnification ECC observation was also performed. In the topmost ~1 μm a nanolaminated structure is developed, as shown in figure 3. Most of the nanolaminated structure is elongated along the direction of the plastic deformation, which is parallel to the surface. The average boundary spacing is not fully resolved in the SEM ECC image, but the thickness scale is approximately 50 – 100 nm. In the depth range from 1-10 μm, alternating layers of ultrafine laminated (UFL) structure and nanolaminated (NL) structure are observed. The ultrafine laminated structures are slightly elongated and have an aspect in the range 2 – 6. The average boundary spacing of the UFL structure is about 130±20 nm. The morphology of each NL regions is similar to that in the topmost 1 μm, but the laminated structure is slightly thicker, with an average boundary spacing of about 105 nm. It is found that the nanolaminated structure in the topmost surface is different from that previously reported in Cu processed by SMAT [1] and SMGT [4, 9], where slightly elongated nanograins are produced in the topmost surface It should be noted that the nanostructures in the gradient layer are generally unstable [11, 12]. The grains may grow either under load or thermal treatments. A substantial temperature rise is unavoidable during the repeated HPSR processing, and this may be high enough to induce noticeable structural coarsening. The formation of the UFL-NL mixed layers in the HPSR processed Cu could be a result of such growth of the nanolaminated structure during HPSR processing.

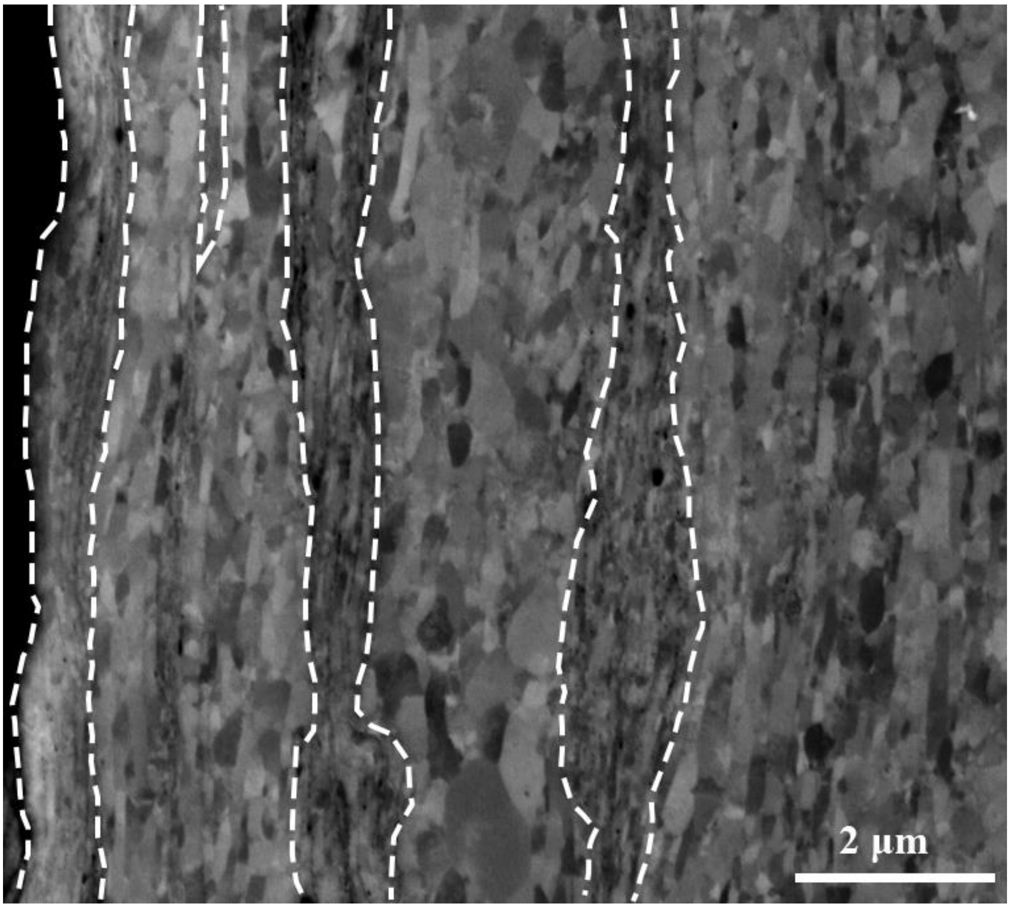

Figure 3. A cross-sectional ECC image of the deformation microstructure in the topmost ~10 μm. The surface is at the left edge. The white dashed line indicates the position of nanolaminated boundaries.

The detailed microstructure in the deformed surface layer was further investigated by EBSD. A gradient microstructure, varying from the topmost surface to the coarse grained bulk is clearly observed in figure 4a-d. The black layer in the left part of figure 4a corresponds to the microstructure in the top ~10 μm in figure 3, where high strain concentration and fine grains make it impossible for EBSD to resolve the details of the microstructure. In depths from 10-300 μm, well-defined nanometer-thick laminated structures parallel to the surface are developed, as shown in figure 4a. The laminated structures are up to several microns in length with thickness ranging from 110±20 nm at a depth of ~20 μm to 270±20 nm at a depth of ~300 μm. A regular lamellar deformation microstructure is observed at depths of 300-600 μm (figure 4b). The boundary spacings vary from hundreds of nanometers to a few micrometers with increasing depth from the surface. As shown in figure 4c, a transition region composed of a lamellar structure and a slightly refined grain layer can be observed at the depths of 600-1200 μm. The right part of figure 4d shows that at depths larger than 2000 μm the grains are slightly deformed but otherwise approximately similar to the annealed microstructure.

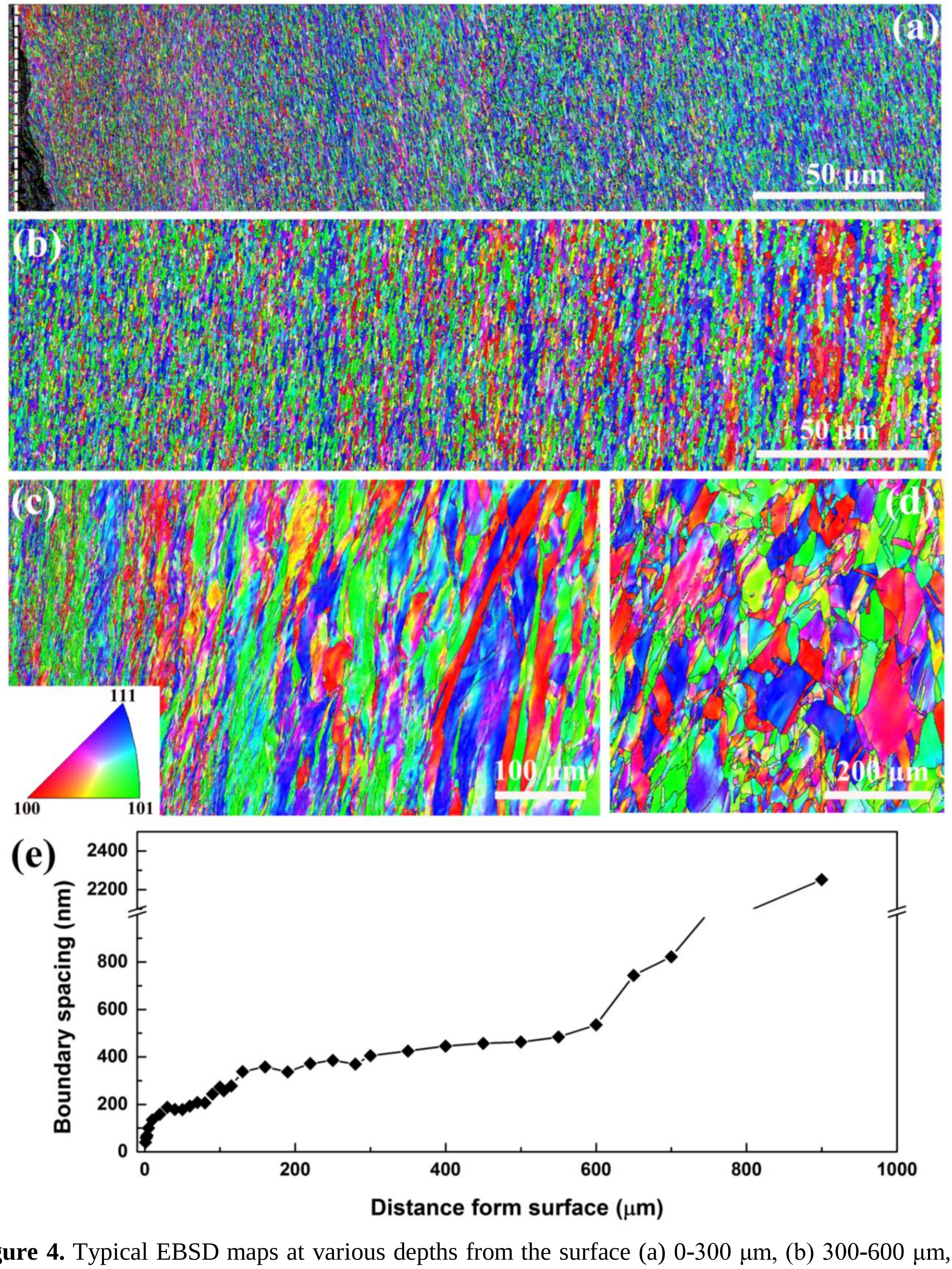

Figure 4. Typical EBSD maps at various depths from the surface (a) 0-300 μm, (b) 300-600 μm, (c) 600-1200 μm and (d) >1200 μm. The white dashed line in (a) indicates the position of the surface. (e) Variation of boundary spacings as a function of distance from the surface, as measured from EBSD maps.

Figure 4e shows the evolution of the boundary spacing as a function of depth. A good accordance is found between the boundary spacing and the hardness results. In the range of depths from 0-150 μm, the boundary spacing increases significantly with increasing depth, corresponding to a relatively steep decrease in hardness. In the depth range of 150-600 μm, a relatively homogeneous ultrafine structure with boundary spacing of about 350±50 nm is developed, which corresponds well to a relatively slow decrease in hardness in this range. At depths larger than 600 μm, the boundary spacing increases with increasing depth, corresponding to a further gradual decrease in hardness. Taken together, the microstructure introduced by HPSR is characterized by a gradient structure from nanostructured grains, to deformed coarse grains, to un-deformed grains, and correspondingly to a gradient of hardness.

Surface mechanical attrition treatment [2], surface mechanical grinding treatment [4, 9], as well as other mechanical treatments [13], have also been used to produce a surface gradient structure in Cu. The refinement of microstructure is attributed to the gradient distribution of strain and strain rate on the sample surface during surface plastic deformation [3]. Although the thickness of the gradient layer is strongly dependent on the materials and treatment parameters, such as rotation speed and tool geometry, the previously reported surface layer thickness values are mostly limited to about 1 mm. However, the present results (figure 2a and figure 4) show that the thickness of the overall deformed surface layer is ~2 mm, which is much thicker than previously reported. In the HPSR process, a load force as high as 20 kN is utilized, which produces a high pressure on the sample surface. Hence, large plastic strains over a broad area and reaching to large depth are achieved during the HPSR deformation, resulting in a thick deformed layer. The HPSR technique provides a new approach for the generation of gradient microstructures extending over a large depth on the surface of bulk metallic materials. This new technique requires only simple procedures and it is expected that the process can easily be scaled-up and adapted to for industrial production and applications.

4. Conclusions

In this study a new technique, high pressure surface rolling (HPSR), has been applied to refine the grain structure on the surface of coarse-grained pure Cu. A deformed surface layer about 2 mm thick is generated, characterized by gradient in grain size, and correspondingly in a micro-hardness gradient, from the surface to the sample interior. The grain size varies from the nanometer scale at the top surface to the micrometer scale in un-deformed interior volumes. The hardness varies from 1.37 GPa near the topmost surface to about 0.6 GPa in the un-deformed coarse-grained matrix. The study demonstrates that HPSR is a promising process for producing a thick layer of gradient microstructure on the surface of bulk metallic materials.

Acknowledgments

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