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Sensitive Copper Detection in P-type CZ Silicon using μPCD

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Conventional microwave photoconductive decay (μPCD) method in conjunction with high intensity bias light has been used to study out-diffusion and precipitation of copper in p-type Czochralski (CZ) silicon. The high intensity light is used to activate the precipitation of interstitial copper, which follows Ham's kinetics and results in a distinctive decrease in the excess carrier lifetime. Results indicate that Cu concentration well below 10^{12} cm^{-3} can be detected by this method. It is demonstrated that positive corona charge can be used to prevent out-diffusion of interstitial copper, while negative charge enables copper to freely diffuse to the wafer surfaces. It was observed that the precipitation rate of copper increased significantly when the bias-light intensity is raised above a certain critical level. In addition, the copper precipitation rate was discovered to be much higher in samples which have internal gettering sites. These findings suggest that (*i*) high intensity light reduces the electrostatic repulsion between positively charged interstitial copper ions and copper precipitates enabling copper to precipitate in the wafer bulk even at a low concentration level, and (*ii*) during high intensity illumination, oxygen precipitates provide effective heterogeneous nucleation sites for copper.

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Copper is a harmful impurity in modern integrated circuit technology; it degrades gate oxide integrity[1] and causes increased junction leakage.[2] To be able to detect copper contamination by contactless minority-carrier lifetime methods is an attractive idea, as measurements are fast, nondestructive, and sensitive to measured defect concentrations, making them a suitable characterization tool for the industrial environment. However, conventional recombination studies show weak dependence of low copper concentration on minority carrier lifetime in p-type silicon.[3,4]

Recently, it was observed by the surface photovoltage (SPV) method that optical activation drastically enhances recombination activity in p-type silicon which contains copper.[5,6] Bazzali *et al.*[7] have reported similar behavior of enhanced recombination activity of copper in silicon in the presence of small oxygen precipitates. By combining these observations, the authors have discovered that the microwave photoconductive decay (μPCD) method in conjunction with high intensity bias light can be used to detect copper contamination in p-type silicon which contains small oxygen precipitates.[8]

One of the objectives of this paper is to gain understanding about the origin of the above-mentioned increase in recombination activity of copper. Previously, it has been observed that in p-type wafers, precipitation of copper takes place in the wafer bulk only when copper concentration exceeds approximately 10^{16} cm^{-3}.[9] This has been explained by the following electrostatic model. As $\mathrm{Cu_i^+}$ ions act as shallow donors in p-type silicon, the Fermi level approaches the conduction band edge (E_C) with increasing copper concentration. When the Fermi level is raised above $E_\mathrm{C} - 0.2$ eV, which is known to be the electroneutrality level of copper precipitates, the charge state of copper precipitates changes from positive to neutral or negative, diminishing the electrostatic repulsion between positively charged interstitial copper ions and copper precipitates. However, copper can precipitate easily in n-type silicon because the Fermi level is close to the electroneutrality level of copper precipitates even at a low copper concentration level.[9] Similarly, we suggest that the electron Fermi level can be raised in p-type silicon by optical activation. This affects the electrostatic repulsion according to the above electrostatic model and results in an enhancement in the copper precipitation rate.

We begin this paper by studying how the light generated excess-carrier density affects the precipitation rate of copper. Then we study the effect of external corona charge on the out- and indiffusion of interstitial copper ions. Finally, we estimate the sensitivity of copper detection of the method used.

Experimental

Sample preparation.—Samples used in this study were single-side polished 100 mm Czochralski grown silicon wafers with 525 μm thickness. The resistivity in boron-doped wafers is 10-20 Ω cm and in phosphorus-doped wafers 3 Ω cm. The initial interstitial oxygen concentration is about 13 parts per million atomic, measured by Fourier transform infrared (FTIR) spectroscopy using the ASTM F 121-83 calibration factor. All wafers were preoxidized at 1050°C for 15 min to dissolve the grown-in precipitate nuclei, *i.e.*, to reduce the effect of the thermal history of the wafer.

Small oxygen precipitates were grown in some of the wafers by a two-step thermal anneal. The nucleation of oxygen precipitates was done in nitrogen ambient at 625°C for 4 h. This was followed by the growth of precipitates in oxygen ambient at 800°C for 4 h. This procedure resulted in an oxygen precipitate density of about 10^9 cm^{-3},[10] which was confirmed by scanning infrared microscope measurements in the corresponding samples with larger precipitate size. The density was small enough that precipitates should not have had an excessive effect on minority carrier recombination by themselves.

Copper was chemically deposited on the oxide-covered wafer surfaces by immersing the wafers in a diluted copper-sulfate solution. Copper indiffusion was performed in nitrogen ambient at 800°C for 20 min through the 17 nm silicon dioxide layer, which was used to diminish unintentional iron contamination. The oxide thickness and the indiffusion temperature used ensured that the copper atoms diffused from the surface of the oxide into the bulk silicon resulting in homogeneous copper distribution throughout the wafer.[11,12]

We also prepared two kinds of reference samples. One set of reference samples had oxygen precipitates but no copper while the other set of reference samples had copper but no oxygen precipitates. In addition, we had wafers which had neither copper nor oxygen precipitates.

Description of measurement method.—In the experiments we use a method, recently developed by the authors, in which the microwave photoconductive decay (μPCD) method is used in conjunction with high intensity bias light.[8] In p-type silicon, the high intensity light activates the precipitation of interstitial copper in the presence of small oxygen precipitates. The precipitation follows Ham's kinetics and results in a distinctive increase in the recombination rate, as copper precipitates have been shown to be very effective recombination centers.[13] The change in recombination lifetime is clearly detectable even when the surface copper concentration is below 10^{12} cm^{-2}.[8]

K. Sunakawa *et al.* have proven that when an electric field is applied to a surface of the silicon wafer, heavy metals existing

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within the bulk of the wafer aggregate to the surface of the wafer.[14] In our study, we use an electric field to prevent out-diffusion of copper in p-type silicon. The electric field was formed by depositing 1 μC/cm^2 positive corona charge on top of the wafer surfaces.[15] If positive corona-charge is omitted, the impact of high-intensity light may be very different. The method is also applicable when the copper has completely out-diffused to the wafer surfaces, as we have demonstrated[8] that it is possible to indiffuse copper back into wafer bulk from the oxide interface by changing the polarity of the surface corona charge from negative to positive. There is no need to optically activate the wafer right after applying the corona charge; we have noticed that the optical activation induces the same amount of decrease in the recombination lifetime even several months after deposition of corona charge.

The minority-carrier recombination lifetime was measured at a high injection level using a standard μPCD technique.[16] A pulsed laser diode of 904 nm wavelength and 200 ns pulse length was used together with a continuous bias light for the excitation of the excess carriers. The wavelength of the bias light was 973.5 nm. The spot size of the pulsed light and the bias light was about 1 mm^2. The density of injected photons per pulse was 1.2×10^{15} photons/cm^2. Carrier recombination after pulsed light induces photoconductance decay, which was recorded by the time-resolved measurements of the microwave power that reflects from the wafer. The frequency of the microwave radiation was 10.04 GHz. The time constant of the measured decay was obtained by a monoexponential fit to the asymptotic photoconductance decay and interpreted as the recombination lifetime.

The intensity of the bias light was manually adjusted by means of an attenuator to maintain excess-carrier concentration of $\Delta n = 10^{16}$ cm^{-3} during the lifetime measurements. It is worth noting that the injection level, *i.e.*, excess carrier concentration, is dependent on the minority carrier lifetime. Therefore, when we wanted to maintain the constant injection level while the minority carrier lifetime decreased, we increased the intensity of bias light accordingly. Based on the measurement repeatability, the measurement error in the recombination lifetime was less than 2% when the lifetime was in the millisecond range and 1% when the lifetime was lower than 500 μs.

Optical activation of copper defects was performed by the same bias-light source, which was used for adjusting the excess-carrier concentration during lifetime measurement. Note that when the lifetime measurement was carried out, the optical activation had to be stopped temporarily in order to reduce the injection to the suitable level (10^{16} cm^{-3}).

The temperature of the sample was measured during the optical activation with a thermocouple attached to the surface of the wafer to make sure the sample did not heat substantially during prolonged optical activation. The sample temperature was 23-27°C during the lifetime measurements and during the prolonged optical activation exposure. Optical activation increased the sample temperature only 3°C at the maximum, which was small enough not to have any significant effect on the measurement.

In the measurements, the effect of optical activation on the minority carrier lifetime was permanent unlike in case of iron-boron pairs. Due to the permanent effect it was not possible to measure the same point more than once. For example, in the copper out-diffusion study, the optical activation had to be done at several separate points on a wafer.

Results and Discussion

Effect of optical activation on recombination rate.—Figure 1 shows an example of the effect of optical activation, with 29 W/cm^2 intensity, on the excess-carrier recombination lifetime in p-type silicon, which had copper concentration about 2×10^{12} cm^{-3}. It can be seen that measured high-injection level lifetime τ (t) decreased from 5 ms to 150 μs during high intensity illumination in a wafer which contained small oxygen precipitates. The decrease in lifetime was much weaker in a homogenized wafer. The reason for this difference is discussed later in this paper.

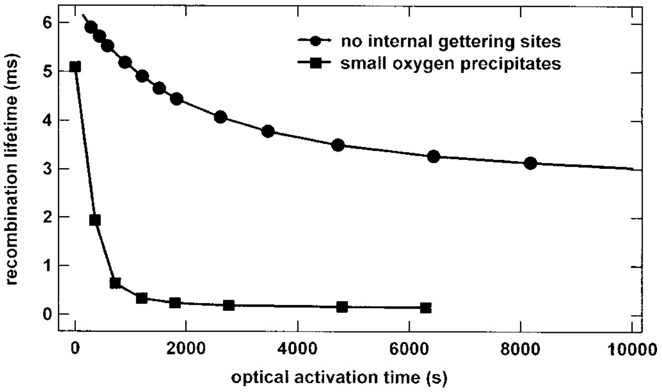

Figure 1. Effect of optical activation on the excess-carrier recombination lifetime in p-type silicon with and without oxygen precipitates. Intentional copper concentration in the samples is about 10^{12} cm^{-3}. Optical activation had no noticeable effect on the recombination lifetime in the noncontaminated reference samples.

Similar measurements of recombination lifetime *vs.* optical activation time were made on reference wafers, which were not intentionally copper contaminated. One set of noncontaminated reference samples had oxygen precipitates and the other set was homogenized. In both sets of the noncontaminated reference wafers, optical activation showed no noticeable effect on the recombination lifetime, *i.e.*, the recombination lifetime remained at the constant level during optical activation. This supports the idea that the observed change in optically activated recombination lifetime is related to the copper.

The measured excess-carrier lifetime $\tau\ (t)$ is converted to optically activated excess-carrier recombination rate $R(t)$ as follows

$$R(t) = \Delta n[\tau^{-1}(t) - \tau^{-1}(0)] \qquad [1]$$

where τ (0) is the measured lifetime without optical activation, *i.e.*, at $t = 0$. Therefore, by applying Ham's law[17] and assuming that the optically activated recombination rate R is proportional to the total amount of precipitated copper, the following equation can be used to fit the experimental data

$$R(t) = R_{\text{final}}[1 - \exp(-t/\tau_{\text{Cu}})] \qquad [2]$$

where τ_{Cu} is the time constant of precipitation. It is determined as follows

$$\tau_{\text{Cu}} = (4\pi n r_0 D)^{-1} \qquad [3]$$

where n is the effective precipitation density, r_0 is the effective radius of the copper precipitates, and D is the diffusion constant of the interstitial copper. It is shown that the obtained value for nr_0 from Eq. 3 is reasonable.[8] Thus, the measured data can be well explained by Ham's law, which gives the precipitation rate of copper as a function of optical activation time.

Effect of optical intensity on recombination rate.—The optically activated recombination rate was measured as a function of optical activation time with different bias-light intensities. The incident optical power was varied between 4.5 and 46 W/cm^2. We determined the time constant τ_0 for each intensity by fitting Eq. 2 to the measurement points. Figure 2 shows both the experimental and fitted data at two different light intensities in a wafer which contains small oxygen precipitates.

It can be seen from Fig. 2 that there was a clear difference in time constants between these two intensities. This result can be explained by different precipitation rates of copper, *i.e.*, in the case of

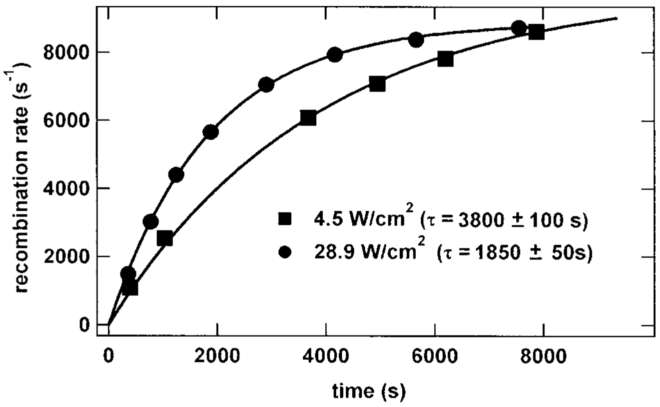

Figure 2. Optically activated recombination rate as a function of activation time at two different incident optical intensities.

higher optical intensity, precipitation of copper is faster and the corresponding time constant is smaller. Figure 2 shows also that the recombination rate saturated close to the same level in the end of the optical activation regardless of the optical intensity. This could indicate that the saturation level is proportional to the copper concentration, because the measurements were made on the same uniformly copper-contaminated wafer.

To further study the influence of the bias-light intensity on the time constant of the optically activated recombination, we found it instructive to calculate the quasi-Fermi levels for electrons with different optical intensities. Calculations were made with commercial PC1D simulation software.[18] To obtain the time constant at different Fermi level positions, we measured the optically activated recombination rate with different optical intensities. Figure 3 shows the results of the obtained time constant of the recombination rate as a function of quasi-Fermi level for electrons E_{fn}.

We can see from the Fig. 3 that, above the equilibrium solubility concentration, the precipitation rate of copper depended on the electron Fermi level. Therefore, it is reasonable to expect that the change in recombination activity was due to electrostatic effects which were affected by the illumination. C. Flink *et al.*[19] have reported that without optical activation, precipitation of copper is very slow in the bulk of the wafer at low copper concentration, mainly due to the electrostatic repulsion between the positively charged copper precipitates and the fast-diffusing interstitial $\mathrm{Cu_i^+}$ ions.[20] However, during illumination, the quasi-Fermi level for electrons rose toward the conduction bandedge, which may have changed the charge state of some defect level(s), thus increasing the recombination activity.

First, we studied the energy range (0.14-0.18) eV to examine if the defect level $E_c - (0.15 \text{ to } 0.16)$ eV, related to the interstitial copper,[21] affects the precipitation rate of copper. Figure 3 shows no significant change in recombination activity around this energy level. The small decrease in the time constant in the energy range (0.14-0.18) eV can be explained by the increase in the effective nucleation site density when the Fermi level is closer to the conduction bandedge.

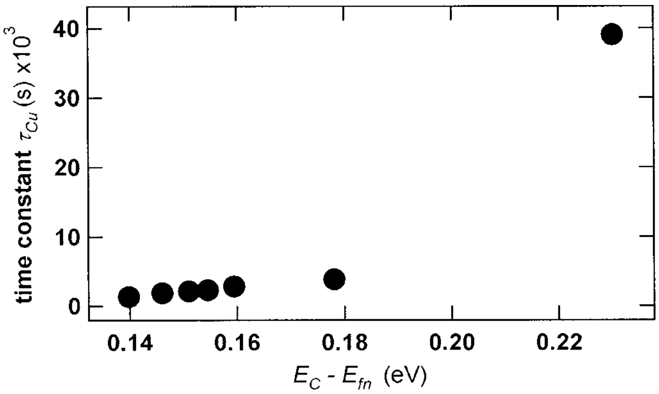

Figure 3. Time constant of the recombination rate as a function of electron quasi-Fermi level in wafers with oxygen precipitates.

Next, copper precipitates and their possible change in charge state during illumination were studied. It is reported that copper precipitates homogeneously forming copper silicides $\mathrm{Cu_3Si}$, which are positively charged with electroneutrality level at approximately $E_c - 0.2$ eV.[20,22] Therefore, we measured the time constant with such a low intensity that the electron Fermi level was well below the electroneutrality level of copper silicides $(E_c - E_{\mathrm{fn}} = 0.23 \text{ eV})$. We observed that the time constant was considerably higher, around 39000 s, which indicates a very low copper precipitation rate. Figure 3 shows that the increase in precipitation rate was very strong between energies (0.18-0.23) eV. This result indicates that there was a significant increase in the copper precipitation rate between energies 0.18-0.23 eV. This supports the idea of the change in the charge state of copper precipitates from negative to positive or neutral.

The same measurements were made on the copper contaminated reference wafers, which went through only the first homogenization anneal, *i.e.*, in those wafers oxygen precipitation density was extremely low. It is seen from Fig. 1 that oxygen precipitates had a significant effect on the precipitation rate of copper. The decrease in the time constant, *i.e.*, the enhancement in the precipitation rate, is explained by the increase in the effective density of precipitation sites (Eq. 3). In samples without intentionally formed internal gettering (IG) sites, copper precipitated slowly, as the effective density of precipitation sites is small. In samples which contain IG sites, the copper precipitation rate was much higher, as there were heterogeneous nucleation sites present. This suggests that oxygen precipitates, indeed, can act as effective sinks for copper.

Although the decrease in lifetime due to optical activation was observed in wafers which did not have intentionally grown IG sites, the samples with IG sites are far more interesting from a practical point of view. If the above method were to be used to detect copper contamination, the required measurement time would be considerably shorter in wafers which had IG sites. Figure 1 shows that the required measurement time to obtain the saturation level was about 30 min in samples with IG sites. The corresponding time in homogenized samples was over 2 h.

Out-diffusion of copper.—When positive corona charge was deposited on both wafer surfaces, the decrease in lifetime after optical activation was the same regardless of the elapsed time after deposition of corona charge. This indicates that the out-diffusion of copper was successfully eliminated by positive corona charge. In contrast, by depositing negative corona charge, the out-diffusion of copper to the wafer surfaces was enabled. By assuming that negative corona charge allows copper to freely out-diffuse to wafer surfaces and using out-diffusion kinetics based on Fick's law, we can estimate the concentration of copper [Cu] in wafer bulk as a function of time

$$[\mathrm{Cu}] = [\mathrm{Cu}]_0 \exp(-t/\tau_{\mathrm{diff}}) \qquad [4]$$

where $[\mathrm{Cu}]_0$ is the initial copper concentration. The time constant of the diffusion, τ_{diff}, is

$$\tau_{\mathrm{diff}} = d^2/(\pi^2 D_{\mathrm{Cu_i}}) \qquad [5]$$

where d is the thickness of the wafer and $D_{\mathrm{Cu_i}}$ is the diffusion constant of the interstitial copper ions.

We studied the out-diffusion of copper by depositing negative corona charge on both sides of the p-type silicon wafer. Then we measured the saturation value of the optically activated recombination rate at constant bias-light intensity (29 W/cm^2) as a function of elapsed time after deposition of corona charge. Because the optically activated recombination rate, measured at the same bias-light inten-

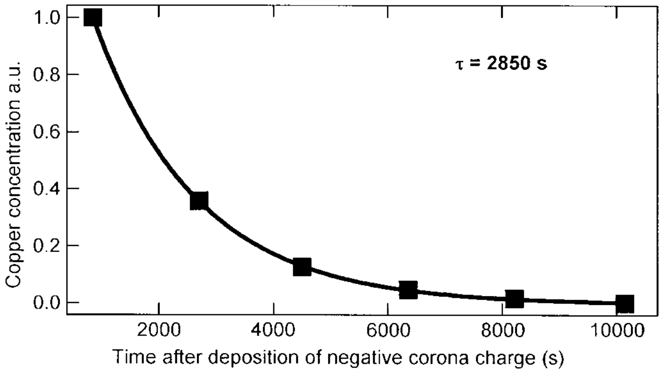

Figure 4. Copper concentration in wafer bulk as a function of time after deposition of negative corona charge.

sity, is proportional to the amount of precipitated copper in the wafer bulk, we got as a result copper concentration (in arbitrary units) as a function of diffusion time. Equation 4 can then be used to fit the experimental data to find the time constant τ_{diff}. Figure 4 presents the experimental results as well as the fitted curve.

The fitted curve was weighted by the last three measurement points; it can be assumed that the copper distribution profile was not necessarily constant immediately after deposition of negative corona. This is because the surface layer was quickly emptied of copper. The obtained time constant of the curve was 2850 s, which corresponds to a diffusivity of 9.8×10^{-8} cm^2 s^{-1}. This is in excellent agreement with the room temperature diffusivity value of interstitial copper in this material (1.3×10^{-7} cm^2 s^{-1}).[23] We have thus shown that the decrease in optically activated recombination rate after deposition of negative corona charge correlates well with the out-diffusion of interstitial copper atoms.

Sensitivity of the method.—We used n-type wafers as a reference to get a rough estimate for the amount of copper in the wafer bulk. It is known that copper reduces the minority carrier lifetime in n-type wafers even in the case of quite low copper contamination level.[5] After intentional copper contamination and in-diffusion, we measured a decrease in the minority carrier recombination lifetime in n-type wafers from 4-5 to 3 ms. This suggests that the copper surface contamination value is less than 10^{12} cm^{-2}.[24]

In order to get a better estimate for the amount of copper in the samples, adsorbed copper concentration at the oxide-covered wafer surface was quantified by vapor phase decomposition total reflection *x*-ray fluorescence analysis using HF-H_2O_2 containing scanning liquid. The total contamination in the oxide and at the oxide/silicon interface was measured. The copper concentration was 1×10^{11} Cu-atoms/cm^2 before indiffusion and 4×10^{10} Cu atoms/cm^2 after the indiffusion anneal. Assuming that no evaporation took place, we calculated that during indiffusion only 60% of the surface copper diffused into the wafer bulk. This resulted in bulk copper concentration of 2.3×10^{12} cm^{-3} in 525 μm thick wafer, which is in fair agreement with the estimate from n-type reference wafers. Figure 1 shows that the lifetime decreased from 5 ms to 150 μs with this 2.3×10^{12} cm^{-3} copper concentration. However, the detection limit was far below this concentration. For example, based on the out-diffusion studies, when less than 10% of the initial copper concentration was left in the wafer bulk due to out-diffusion, the decrease in lifetime is still clearly detectable. Thus, we suggest that copper concentration in the range of 10^{11} cm^{-3} can readily be detected by this method. However, we emphasize that in case some of the copper had already precipitated in the wafer before optical activation, we could not detect total copper concentration until all copper was first redissoluted in high temperature. To obtain the exact sensitivity of this method, other independent methods should be used to determine the quantitative amount of copper present in samples.

Conclusions

Recent studies of copper precipitation and out-diffusion in p-type silicon have shown that below a critical contamination, the copper predominantly diffused out to the wafer surface, while for higher copper concentrations, it mainly precipitated in the bulk.[9,19] The behavior is explained by the electrostatic interaction between the positively charged interstitial copper and the copper precipitates. We have shown that copper can precipitate in wafer bulk far below this critical concentration under high intensity bias light and that positive corona charge can be used to prevent its out-diffusion on thermally oxidized wafers.

In addition, studies of internal gettering of copper have indicated that oxygen precipitates are relatively inefficient as sinks for copper.[25] However, our experiments show that under high-intensity illumination, oxygen precipitates provided effective heterogeneous nucleation sites for copper. This could find application in developing gettering for copper.

It has been reported that surface band bending repulses positively charged copper ions, which retards the out-diffusion of copper.[19] We have shown that when negative corona charge was deposited on p-type silicon wafer the copper ions could diffuse freely to the wafer surfaces.

Based on the findings of this work we have demonstrated a promising and easily applicable method to detect low copper concentrations in p-Si. We estimate that a copper concentration of about 10^{11} cm^{-3} can be detected by this method. To be able to use this method as a quantitative method for determining the copper concentration in p-type silicon wafers, the measurement results should be calibrated with some other quantitative method, for example transient ion drift.[26]

As Ham's kinetics describes well copper precipitation, we expect that the precipitation rate is dependent on oxygen precipitate size and density. We will apply this technique in the future for more detailed studies of the role of oxygen precipitates to get some data related to intrinsic gettering efficiency. In addition, more work concerning the copper concentration sensitivity will be performed.

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