Investigation of the bonding strength and interface current of *p*-Si/*n*-GaAs wafers bonded by surface activated bonding at room temperature

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Equivalent bulk strength of the interface between p-Si/n-GaAs bonded through the surface activated bonding (SAB) method is found. The interface current was extensively investigated. Nonideal behavior of the pn junction current is found to be due to the tunneling current between the conduction band and valence band across the transition region associated with band gap states. Interface current decreases with increasing sputtering time and energy and vice versa. Irradiation time and energy dependent behavior indicates that the accumulation of radiation induced defects associated with the doping controls the interface current of p-Si/n-GaAs. Moreover, strong impact of the exposure to an ultrahigh vacuum atmosphere of the activated surfaces on the interface current of p-Si/p-Si is found. Finally it can be suggested that a laser diode can be fabricated by the bonding between p-Si and n-GaAs through the SAB method, because of the achievement of equivalent bulk strength of the interface. © *2001 American Vacuum Society.* [DOI: 10.1116/1.1414115]

I. INTRODUCTION

Fabrication of light emitting semiconductor diodes and laser devices requires high quality interfaces among semiconductor layers. The integration of Si and GaAs is promising for such applications. However, single crystal epilayers of GaAs have been found to be difficult to grow due to the lack of an appropriately lattice matched substrate material Si with a reasonably close thermal expansion coefficient. Further complications arise from the structural differences where Si is the diamond structure and GaAs is the cubic zinc-blende type material. A relatively new bonding technique, called surface activated bonding (SAB), has emerged to circumvent these demerits. SAB is a solderless bonding method that joins two similar and/or dissimilar clean surfaces by means of the adhesive force of surface atoms in an ultrahigh vacuum (UHV) at room temperature (RT).[1] The clean surfaces are prepared by sputtering the sample surfaces with an argon fast atom beam (Ar-FAB) in the vacuum condition.

The performance of the bonding interface of relevant mating semiconductors produced by the SAB method must be demonstrated before the fabrication of the optical device. The performance should be evaluated as a function of the surface flatness, the bonding strength, and the pn junction quality. Hosoda and her colleagues have reported that the higher the surface roughness, the lower the bonding strength of Al and Al_2O_3 bonded through the SAB process.[2] They have also investigated the influence of the exposure to an UHV atmosphere of the surfaces of Al, Si, and SiO_2 activated by the Ar-FAB on the bonding strength.[3] Moreover, since the mating surfaces are cleaned with a high energetic (0.6–1.5 keV) Ar-FAB source, the effect of irradiation on the interface current must be demonstrated. In fact, while there have been a number of reports on the SAB of Si/Si,[4] Si/InP,[5–7] and Si/GaAs,[5–7] little has been reported on the effect of exposure to an UHV atmosphere of the surfaces activated by the Ar-FAB on the bonded interface current. Electrical characterization, especially current–voltage (I–V) characteristics, can provide precise information on the state of the bonded interface.

In this article we report on the bonding of p-Si and n-GaAs and the evaluation of the bonding strength as well as interface state through the tensile test and the I–V measurements, respectively, under various conditions of sputtering as well as exposing in an UHV atmosphere of the surfaces.

II. EXPERIMENTAL PROCEDURE

A. Sample preparation

Mirror polished samples of p-Si(100) and n-GaAs(100) having dimension of $(5\times5\times0.45)$ and $(10\times10\times0.35)$ mm^3, respectively, were used. The resistivity of p-Si and n-GaAs was in the ranges of 0.01 to 0.02 and $(2.2\text{ to }2.3)\times10^{-3}\,\Omega\,\text{cm}$, respectively. Doping elements for p-Si and n-GaAs were B and Si, respectively, with the respective carrier concentration of 5×10^{18} and $(1.2\text{ to }1.3)\times10^{18}\,\text{cm}^{-3}$. The mesa structured Si was used to avoid any effects of the edges of the small sample on the bonding. Therefore the contact area between bonded samples was (3×3) mm^2. The p-Si sample was cleaned with H_2SO_4 (4) and H_2O_2 (1) solution at 65 °C for 10 min followed by immersion in 3% HF to remove oxide layers. The sample was then boiled in a solution of NH_4OH (1), H_2O_2 (1), and H_2O (5) at 65 °C for 10 min. The sample was rinsed in water after each chemical treatment. Finally, the p-Si sample was dried by blowing nitrogen on the surfaces. The n-GaAs sample was cleaned with acetone and ethanol only.

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B. Bonding procedure

The SAB machine used for this research consists of six UHV chambers: two preparation, a processing, a transportation, an analysis and a bonding chamber. Detailed information regarding the SAB apparatus can be seen elsewhere.[3] The chemically cleaned samples were loaded to the load lock chamber of the SAB machine, and we waited until the vacuum pressure of the chamber lowered to $\sim 10^{-6}$ Pa. The samples were then transferred to the processing chamber and cleaned separately by sputtering either with a 0.6 or a 1.5 keV Ar-FAB ion dose rate of $2.38\times 10^{14}\, i/\mathrm{cm}^2\,\mathrm{s}$ in the processing chamber for 15–600 s. The beam current used for sputtering in both energies was 15 mA. All of these activities are done in an UHV pressure. The active surface can be dirty with elapsing time even at an UHV of 10^{-7} Pa, so the sputtered samples were transferred to the bonding chamber as quickly as possible, then brought into contact, and finally the bonding was performed with a load of 40 kgf at RT. The total time required for both samples from sputtering to the bonding including transportation was about 23 min.

III. RESULTS AND DISCUSSION

A. Surface characterization

Elemental distribution of sample surfaces was characterized by Perkin-Elmer x-ray photoelectron spectroscopy (XPS) using a monochromatic Mg $K\alpha$ x-ray radiation source at 15 kV and 400 kW. Figure 1(a) shows the XPS spectra of p-Si before (lower curve) and after (upper curve) sputtering. Figure 1(b) shows the XPS spectra of n-GaAs before (lower curve) and after (upper curve) sputtering. The energy of Ar-FAB used for sputtering for both samples was 1.5 keV. The sputtering time for p-Si was 60 and 180 s, while the sputtering for n-GaAs was 180 s. The spectra of sputtering samples were taken subsequently to sputtering. Strong peaks for carbon and oxygen are observed on both sample surfaces before sputtering. The peaks for carbon and oxygen in both samples disappeared after sputtering, indicating active surfaces.

Surface smoothness is an important parameter of the SAB process that can strongly affect the bonding performance, especially in the case of bonding without load. An additional parameter is the surface flatness that absorbs the mechanical stress by distributing all over the area during the SAB process if the samples are very thin. Measurements of surface roughness were carried out by an atomic force microscope (AFM) from Seiko Instruments. Figure 2 shows the time dependence of sputtering on the surface roughness of Si and GaAs with a 0.6 Ar-FAB. The root mean square (rms) value of the surface roughness of both Si and GaAs before exposing Ar-FAB was 0.2 nm. On the other hand, the surface roughness of GaAs was increased to 0.6 nm after sputtering for 600 s, while the rms value remained constant for Si. In both samples a variation of 2% to 3% in the rms values was observed. In the case of 1.5 keV, higher rms values are expected. However, the AFM studies reveal that the surface of the GaAs sample sputtered with an Ar-FAB source gets rougher.

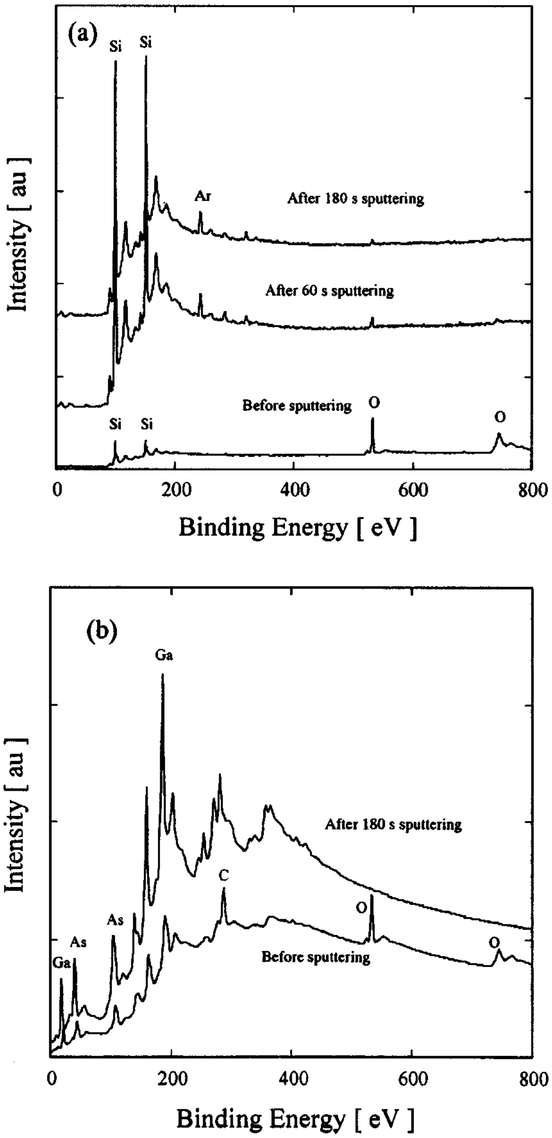

FIG. 1. XPS spectra of (a) p-Si and (b) n-GaAs surfaces before and after sputtering with a 1.5 keV Ar-FAB dose rate of $2.38\times 10^{14}\, i/\mathrm{cm}^2\,\mathrm{s}$ in an UHV at RT. The sputtering time for Si was 60 and 180 s and for GaAs was 180 s.

B. Bonding strength

The optoelectronic devices fabricated through any bonding process must have a certain level of bonding strength at the interface in order to withstand mechanical shocks. We have performed tensile tests for all sets of samples sputtered for various times in order to evaluate the bonding strength. The tensile strength is expressed by $\sigma_{\mathrm{TS}}=F_{\max}/A$, where $F_{\max}$ is the maximum tensile load and A the bonded area. The bonded areas are measured by the use of an optical microscope after a fracture test. Figure 3 shows a typical picture of the broken joints of p-Si and n-GaAs by a tensile test. Figures 3(a) and 3(b) show the broken sides of Si and GaAs, respectively. Most of the samples were broken from the bulk but not from the bonded interface. No significant variation in the bonding strength sputtered for various times was found. The estimated average bonding strength of Si/GaAs was 0.6

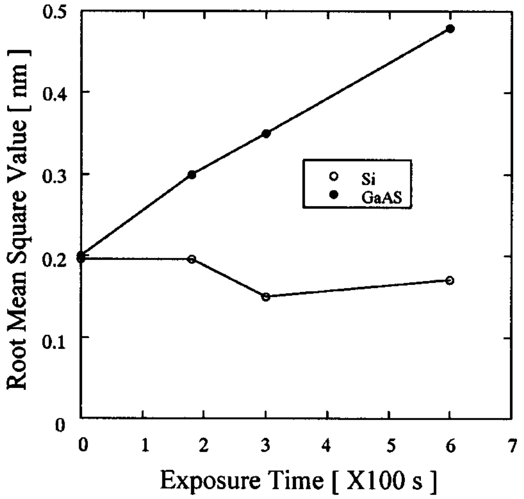

FIG. 2. Time dependence of the sputtering on the surface roughness of p-Si and n-GaAs with a 0.6 Ar-FAB. Measurements were carried out by an AFM at RT and the rms values were plotted.

MPa. This fact indicates that there is no correlation between the bonding strength and the surfaces roughness of Si/GaAs, which is inconsistent to that of Al/Al_2O_3.[3] Anyway, the bonding strength of Si/GaAs is enough for handling the optoelectronic applications.

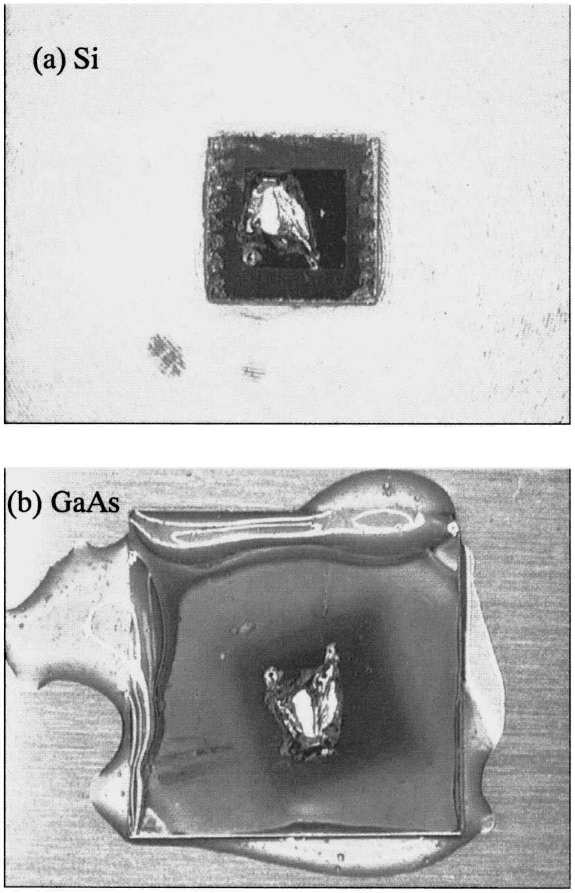

FIG. 3. Bulk fracture image of the p-Si/n-GaAs interface bonded by the SAB method at RT.

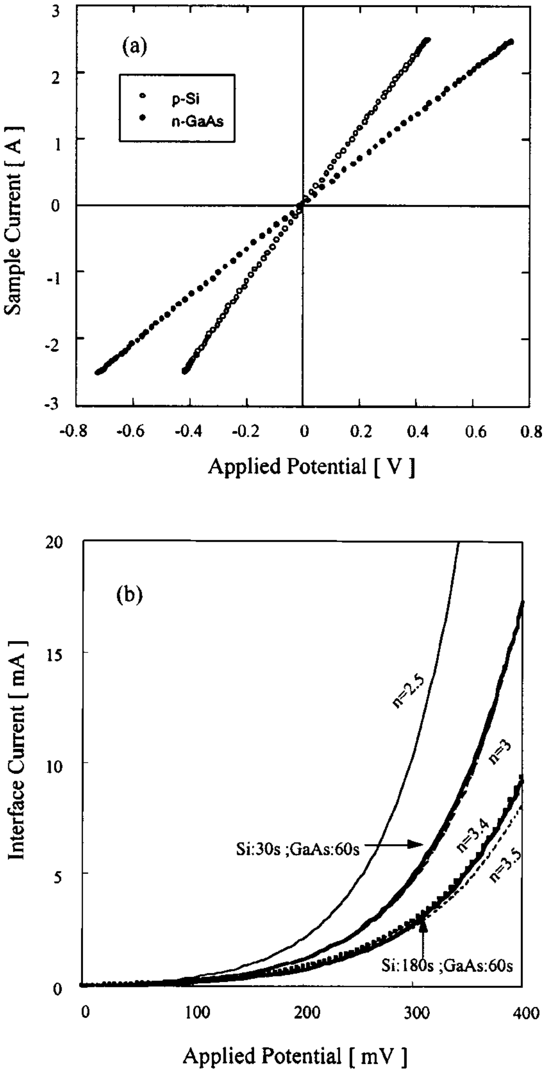

FIG. 4. (a) $I-V$ characteristics of p-Si and n-GaAs before the bonding experiment. (b) Comparison of the theoretical and experimental $I-V$ characteristics of the p-Si/n-GaAs junction bonded by SAB at RT. Experimental results include the data for the interface current of Si/GaAs samples sputtered with a 1.5 keV Ar-FAB for 30 (Si) and 60 (GaAs) s, and 180 (Si) and 60 (GaAs) s.

C. Comparison of theoretical and experimental *I*–*V* characteristics

The electrodes for electrical measurements were made before the bonding experiments in order to protect the bonded interface from the heating effect that is usually required for ohmic contact. A top and a bottom Au electrode of 3 mm diameter each was deposited on the p-Si sample. On the other hand, similar sizes of electrodes were made using Au/Ge on the n-GaAs sample and annealed at 673 K for 60 s. The heating rate was 150 K/min. Ohmic behavior was achieved in both samples before the bonding experiment [Fig. 4(a)]. The $I-V$ characteristics were measured by a Tektronix programmable curve tracer (370A). Generally, the pn junction of an ideal diode can be expressed by $I = I_o(e^{qV/nkT}-1)$,[8] where n is the ideality factor that determines the departure from the ideal characteristics. The value

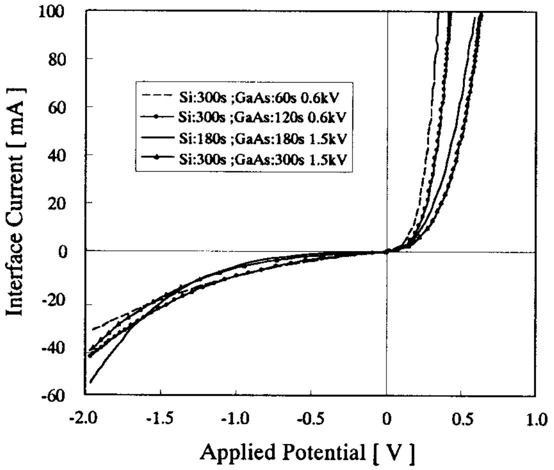

FIG. 5. Time and energy dependence of the Ar-FAB on the interface current of *p*-Si and *n*-GaAs bonded by SAB at RT. In the case of 0.6 keV Ar-FAB, *p*-Si and *n*-GaAs samples were sputtered for 300 (Si) and 60 (GaAs) s, and 300 (Si) and 120 (GaAs) s. For 1.5 keV Ar-FAB, *p*-Si and *n*-GaAs samples were sputtered for 180 (Si) and 180 (GaAs) s, and 300 (Si) and 300 (GaAs) s. The dose rate in both cases is 2.38×10^{14} i/cm^2 s.

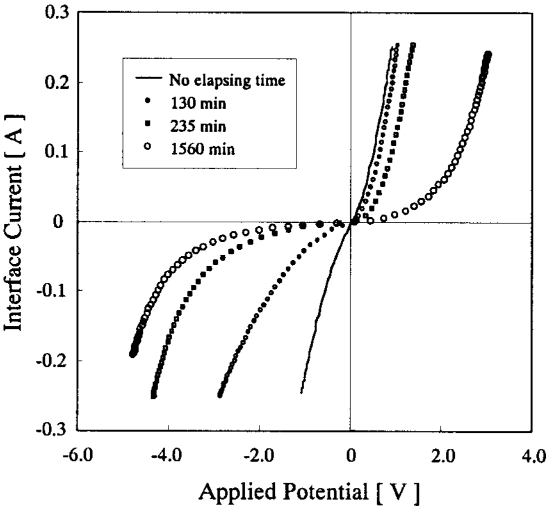

FIG. 6. Time dependent exposure effect to an UHV atmosphere of the activated surfaces by the sputtering of an Ar-FAB on the interface current of *p*-Si/*p*-Si. The samples were held for 0, 7800 (2.6 h), 14 100 (3.9 h), and 93 600 (26 h) s at 2×10^{-7} Pa after sputtering for 180 s with a 1.5 keV Ar-FAB source dose rate of 2.38×10^{14} i/cm^2 s.

of n varies between 1 and 2, depending on the material and temperatures. Other symbols in the equation have their usual meanings.

The experimental and the theoretical $I-V$ characteristics of the *pn* junction must be compared to evaluate the quality of the bonded interface. The theoretical calculation is based on the assumption that $I_o = 2\times 10^{-5}$ A. Figure 4(b) shows the comparison of the theoretical and the experimental $I-V$ results for Si/GaAs, which indicates that the experimental *pn* junction behavior is far from the ideal diode. One can see that the experimental results for Si/GaAs sputtered for different times fit closely to the theoretical curves with the n values varying from 3 to 3.5. The nonideality behavior of the Si/GaAs *pn* junction can be attributed to the electron tunneling between the conduction band and valence band across the transition region through the band gap states.[9]

D. Influence of time and energy of sputtering source on the interface current

The influence of sputtering time and energy of Ar-FAB on the interface current of *p*-Si and *n*-GaAs is shown in Fig. 5. The $I-V$ curves include the results of the interface current of the samples sputtered with 0.6 and 1.5 keV Ar-FAB for various times. In the case of 0.6 keV Ar-FAB, the *p*-Si and the *n*-GaAs sample surfaces were sputtered for 300 (Si) and 60 (GaAs) s and 300 (Si) and 120 (GaAs) s. For 1.5 keV Ar-FAB, the *p*-Si and the *n*-GaAs samples were sputtered for 180 (Si) and 180 (GaAs) s, and 300 (Si) and 300 (GaAs) s. One can easily notice from Fig. 5 that the interface current decreases with increasing sputtering time. Although the sputtering time of the sample surfaces treated with 0.6 keV is higher than that of the first set of sample surfaces treated with energy 1.5 keV, more current flows in the former bonded interface both in the forward and reverse bias condition. These results also show a substantial effect of Ar-FAB energy on the interface current, which indicates that higher energy produces more damages than those of lower energy. Generally, the defects increase with increasing beam energy and irradiation time.[10] So the accumulation of sputtering induced defects results in an amorphous layer[5–7] across the bonded interface and it may control the interface current associated with the sample impurities. In addition, a leaky behavior of the interface current is apparent at the reverse bias condition, which is most likely to be due to the carrier generation in the transition region. Recently we have found an identical sputtering time as well as energy effects on interface current of Si/InP irradiated under 0.6 and 1.5 keV for 30–600 s.[11]

E. Effect of exposure to an UHV atmosphere on the interface current

Although the vacuum pressure in all chambers of the SAB machine is better than 2×10^{-7} Pa, there are still residual gas molecules/atoms such as H_2, H_2O, CH_4, N_2, C, etc. in an UHV atmosphere of the chambers.[3] As previously mentioned, higher elapsing time for transferring samples from processing to bonding stages, hinders bonding of the samples. So the time dependence of the residual particle's effect on the bonding quality requires investigation. Figure 6 shows a typical example of the time dependent residual gas behavior of the interface current of *p*-Si/*p*-Si. The reason for using *p*-Si/*p*-Si samples instead of *p*-Si/*n*-GaAs pairs for these experiments was due to the lesser time and easier process for fabricating ohmic electrodes on *p*-Si than on *n*-GaAs. The experiment consists of sputtering the samples for 180 s, holding the samples in the bonding chamber for various times after sputtering, and finally performing the bonding experiment. Electrical measurements of the interface current were done in the same way as mentioned in the previous section. The samples were held for 0, 7800 (2.6 h), 14 100 (3.9 h), and 93 600 (26 h) s. Alternately, the exposure amount was 0, 12, 21, and 65 L (where L is the abbreviation for Langmuir, which represents a value of 1.33×10^{-4} Pa s). As can be seen in Fig. 6 the interface current decreases with increasing the exposure time in an UHV atmosphere in the bonding chamber. The results indicate that even though the samples elapsed for 26 h (65 L) are bonded, the interface resistivity is very high, which is very bad for any device application. The reason for increasing the resistivity of the Si/GaAs interface with increasing the exposure time seems to be identical to the reason for decreasing the bonding strength of Al/Si and Si/SiO_2 with increasing the exposure time in an UHV atmosphere.[3] Therefore the increased resistivity can be explained in terms of the deposition of residual gas atoms on the surfaces, which produces impurity states and traps for electrons, thereby reducing the interface current.

IV. PERSPECTIVE

The performance of the interface of p-Si/n-GaAs bonded by the SAB process was thoroughly investigated. The bonding strength and electrical resistivity of the interface are found to be excellent for the fabrication of optoelectronic devices like a laser diode. In particular, the mounting of a GaAs-based laser structure is planned on the Si substrate by the SAB process. Recently, we have fabricated an InGaAs/InP on GaAs by the SAB process at RT. Excellent performance is demonstrated by comparison with the as-grown laser device manufactured by metalorganic chemical vapor deposition.[5]

V. CONCLUSIONS

Equivalent bulk strength of the interface between p-Si/n-GaAs bonded through the SAB method is found. Interface current decreases with increasing sputtering time and energy. Irradiation time and energy dependent behavior indicates that the accumulation of radiation induced defects associated with the doping controls the interface current of p-Si/n-GaAs. Strong influence of the exposure to an UHV atmosphere of the activated surfaces on the interface current of p-Si/p-Si is found to be due to the deposition of residual atoms on the surfaces, which produces impurity states and traps electrons, thereby controlling the interface current. Finally it can be suggested that the laser diode can be fabricated by the bonding between p-Si and n-GaAs through the SAB method because of the achievement of equivalent bulk strength of the interface.

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