Review Article

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Study on the preparation process and sintering performance of doped nano-silver paste

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Abstract: After tin–lead solders are banned, the widely used electronic packaging interconnect materials are tin–silver, tin–copper, and other alloy solders. With the application of high-power devices, traditional solders can no longer meet the new requirements. Nano-silver (Ag) paste, as a new solder substitute, exhibits excellent properties, such as excellent thermal and electrical conductivity, sintering at lower temperatures, and service at high temperatures. However, many organic devices still cannot withstand this temperature, and are often not suitable for the connection of nano-Ag paste packaging materials, therefore, it is very urgent to further reduce the sintering temperature of nano-silver paste. Based on the transient liquid phase sintering technology, by doping the nano-Ag paste with the nano-tin paste with a lower melting point to make the two uniformly mix, pressureless sintering at low temperature can be realized, and a sintered joint with a connection strength greater than 20 MPa can be formed. Considering that tin is easy to be oxidized, and the core–shell material can prevent the oxidation of tin, and at the same time ensure the uniform distribution of tin in silver, the doping scheme of the core–shell structure is determined. Heterogeneous flocculation method refers to the particles with different properties of charges which attract each other and agglomerate. It is a continuous reduction method for preparing core–shell materials. This method has the advantages of mild reaction conditions and less equipment investment, so the heterogeneous flocculation method is selected to prepare Sn@Ag core–shell nano paste. And research its sintering performance and strengthening mechanism.

Keywords: nano-silver paste, transient liquid phase sintering, heterogeneous flocculation, tin doping, strengthening mechanism

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1 Development of interconnection materials for electronic packaging

In the past few decades, with the development and improvement of silicon carbide semiconductor technology, more and more power devices that can withstand high frequency, high-temperature, and high-pressure conditions are used in power electronic conversion device of high-speed rail, wind power, solar power, and high-power motors. Electronic packaging, as a technology to realize the electrical interconnection of the chip and the external circuit, not only provides mechanical fixation, sealing protection, and heat dissipation protection for the chip but is also a bridge of signal transmission between the chip and the external circuit. Therefore, the reliability of electronic packaging has a direct impact on the performance of the chip and the device composed of the chip. The characteristics of silicon carbide semiconductors and the use environment of power devices have increased the requirements for chip packaging. Therefore, a large number of researchers are working to find packaging technologies that can be applied to high temperature, high pressure, and high frequency conditions, so that the advantages of silicon carbide semiconductors are fully released.

At present, the development of microelectronic interconnection technology is quite rapid, and the requirements for its reliability are becoming more and more strict. Factors such as the form of interconnection and the specific composition of solder will affect the effect of the final package. The electronic chip is connected to the substrate by solder, and the performance of the interconnect solder is generally viewed from the aspects of conductivity, heat dissipation, and long-term effectiveness [1]. The widely used chip connection materials are alloy solder [2] and conductive glue [3].

For a long time, tin–lead (Sn–Pb) solder was a kind of interconnection solder commonly used in the semiconductor industry. Because of the great harm to the human body, the European Union banned the use of Sn–Pb solder in 2006. Pb-free in the semiconductor industry gradually represents the trend of the world. Sn–Pb solder has a wide range of resources, and has many advantages such as low melting point, good thermal conductivity and plasticity, and good reliability. Therefore, finding new materials after Sn–Pb solder has become the focus and difficulty in the industry [4]. Judging from the current situation, the vast majority of alloy solders contain Sn elements, commonly used are SnAu, SnAg, SnAgCu, etc. In addition, Bi, In, Zn, Sb, Ge, and other elements have also received extensive attention [5]. The eutectic point temperature and eutectic composition of Sn-based solder are shown in Table 1 [5].

Tin–zinc solder has been widely noticed because its melting point is very similar to that of Sn–Pb solder. The advantage of zinc is that it is abundant in resources and low in cost, but zinc is easily oxidized and has poor wetting properties. The wettability of bismuth is good, but bismuth is a derivative product of lead extraction, so the use of bismuth is limited. Due to its strong activity, germanium is contained in many solder components. Indium is easily oxidized, and the content of germanium and indium on earth is very small, so the high cost limits their application range [5].

The Sn-based solder and the copper base plate will form an intermetallic compound (IMC) Cu_6Sn_5 to create a mechanical and conductive connection. Unlike Sn–Pb solder, Pb-free solder forms a coexistence of IMC and tin. $AuSn_4$ can be formed in Sn–Au solder, which means that the molar ratio of Au–Sn in the alloy is 1:4, so less gold can produce a lot of IMC, but $AuSn_4$ is harder and brittle, so the content of the Au in Au–Sn solder should not exceed 5 wt%. The multi-component solder SnAgCu (SAC) can generate a high-strength interconnection with the copper base plate, and its heat dissipation and connection strength are better than Sn–Pb solder. The melting point of the eutectic compound of SAC solder is 217°C, which limits its use range. Due to the low adaptation temperature of some substrates, most of the flux will be evaporated at 217°C, and the solder easily forms gaps at high temperatures. With the passage of time, the gaps will spread outside, reducing the reliability of solder joints.

Table 1: Melting point of Sn-based Pb-free solder [5]

System	Eutectic temp (°C)	Eutectic composition (wt%)
Sn–Cu	227	0.7
Sn–Ag	221	3.5
Sn–Au	217	10
Sn–Zn	198.5	9
Sn–Pb	183	38.1
Sn–Pb	139	57
Sn–In	120	51

Resin conductive adhesive is widely used in electronic packaging of devices due to its low curing temperature and simple manufacturing process. The conductive adhesive with thermosetting polymer as the base material is suitable for service in higher thermal conditions, so the base material of the conductive adhesive is selected as a thermosetting polymer. There are many epoxy functional groups in the polymer, because of the presence of active epoxy functional groups, the polymer will form a cross-linking reaction with various curing agents [6]. Epoxy resins are categorized into three types according to their reaction types: homocluster, latent type, and addition type [7]. However, in general, the electrical conductivity and thermal conductivity of thermosetting polymers are not good, so the metal content in the conductive adhesive can be as high as 80%. In the conductive adhesive, current paths are generated due to the point contact between metal particles, as shown in Figure 1 [8]. Marcq et al. [7] and others used multi-component fillers to mix micron silver powder and carbon nanotube stacked-cup carbon nanotube (SCCNT) into epoxy resin. The interaction between micron silver powder and SCCNT greatly improved its conductivity and connection strength.

The disadvantages of Pb-free alloy solders and conductive adhesives are lower service temperature and lower electrical conductivity and thermal conductivity. The service temperature of alloy solder should be lower than its melting point, and its thermal conductivity is lower than that of pure metal. The service temperature of conductive adhesive is often below 120°C, and the thermal conductivity is lower than that of alloy solder. Therefore, conductive adhesives are generally only used

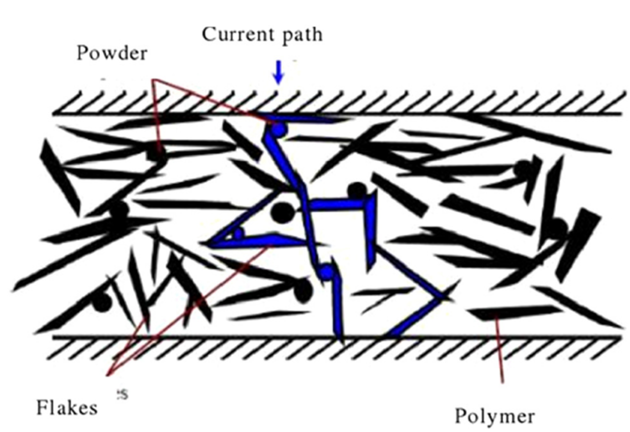

Figure 1: Schematic diagram of anisotropic conductive adhesive [8].

in electronic packaging with lower power. These two traditional solders can no longer adapt to the environment of high-power packaging systems. At the same time, the use of silicon carbide chips is gradually increasing compared to ordinary silicon chips. The service temperature of silicon carbide chips can reach 600°C, and the thermal conductivity of silicon carbide is higher than that of silicon. Silicon carbide chips have higher requirements for connection solder, and Pb-free alloy solder and conductive adhesive obviously fail to meet the performance requirements. Therefore, it is very urgent to find a new interconnection solder.

2 Research status of nano-silver paste

As a kind of electronic packaging interconnection material that can replace traditional solder, nano-silver (Ag) paste has been extensively studied. The high thermal conductivity of bulk Ag can meet the heat dissipation requirements of high-power packages. In addition, due to the nanometer effect, nano-Ag has a lower melting point and can be sintered together at a lower temperature [9]. Therefore, nano-Ag paste can obtain interconnection joints at a lower sintering temperature, and the remelting temperature after sintering is higher, and it can be used at a higher temperature. This performance is suitable for multi-level packaging systems, so that the use range of the nano-Ag paste is expanded [10].

When the silver powder in the silver paste is nano-Ag particles, the silver paste is collectively referred to as nano-Ag paste. Realize the diffusion and sintering of nano-Ag paste at a lower temperature, so as to achieve the purpose of connecting electronic components and substrates [11]. The sintering temperature of nano-Ag paste is usually about 30% of the melting point of silver, and the solder joint composition after heating and sintering is more than 95 wt% silver [12]. Therefore, the solder joints can well inherit the high electrical conductivity, high thermal conductivity, and high melting point (962°C) of silver. These characteristics are very important and beneficial to the power chip packaging system. The sintering temperature of traditional micron silver paste is usually above 600°C, and it is necessary to add lead oxide and other substances harmful to the environment and human body. However, the sintering temperature of nano-Ag paste is generally below 300°C and does not contain substances harmful to the human body and the environment, which can better realize energy conservation and environmental protection. Therefore, nano-Ag paste has received a lot of attention as a promising alternative to traditional solder for silicon carbide chip packaging materials [13,14].

Du et al. [15] used multi-scale silver paste containing nanoparticles and particles to bond the chip on a bare copper substrate under air conditions. After sintering, the oxide layer generated between the sintered Ag and the copper substrate is composed of Cu_2O and Cu. The analysis of the lattice structure at the interface between the sintered Ag and the oxide layer clearly confirmed the formation of the Ag–Cu_2O chemical bond and the Ag–Cu metal bond.

Zhang et al. [16] studied the interface, sintered structure, and shear strength of Si/SiC chip connection through scanning electron microscope, transmission electron microscope, and shear test. The results show that the sintered Ag layer has a porous structure and adheres closely to the electroless Ni–Au surface of the DBC substrate to form a continuous Ag–Au interdiffusion layer. When the sintering pressure is 10 MPa, the shear strength of Si and SiC chip attachments is higher than 35 MPa.

Ide et al. [17] selected Ag powder with a size of about 11 nm to prepare nano-Ag paste with myristyl alcohol as a dispersant, as shown in Figure 2. When subjected to a sintering pressure within 5 MPa and heated to a temperature of 300°C, the connection of copper and copper was achieved after sintering with nano-Ag paste as the interconnect material, and the sintered joint was relatively dense (as shown in Figure 3). The team also used different Ag pastes with a size of about 11 and 100 nm for comparative analysis, and found that the smaller size of the nano-Ag paste would cause damage in the sintered silver layer, as shown in Figure 4, while the larger size of the nano-Ag paste basically breaks at the connection between the Ag layer and the Cu, as shown in Figure 5. In order to obtain higher connection strength at lower temperature, it is necessary to withstand greater sintering

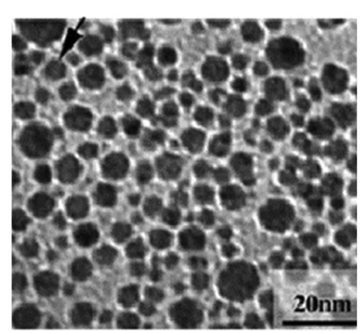

Figure 2: TEM image of Ag.

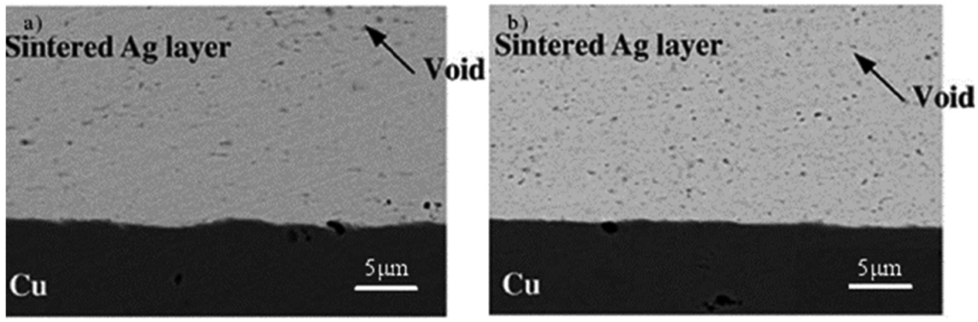

Figure 3: SEM images of Ag and copper layer under various pressures.

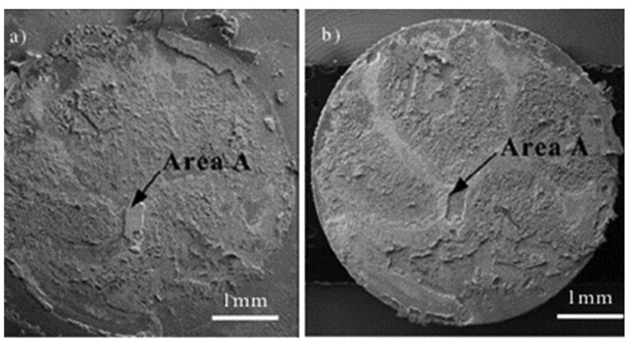

Figure 4: SEM image of fracture of small-sized Ag paste sintered joint.

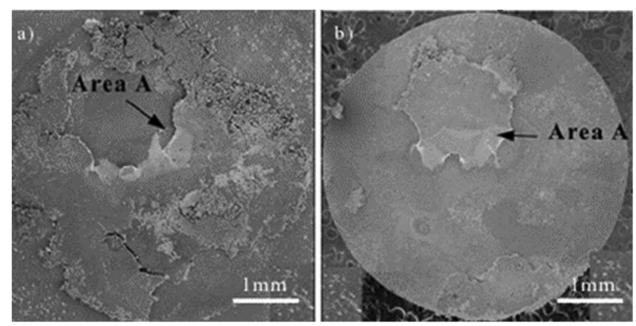

Figure 5: SEM image of fracture of large-sized Ag paste sintered joint.

pressure in the interconnection process, and the interconnection strength at this time can reach 20 MPa.

3 Development trends of doped nano-Ag paste

However, the sintering temperature of nano-Ag paste is not perfect for industrial applications. Many organic devices cannot withstand higher temperatures and are often not suitable for the connection of nano-Ag paste packaging materials. Therefore, for the industrial application of electronic packaging materials, it is very urgent to further reduce the sintering temperature of nano-Ag paste. Generally, in scientific research, the commonly used method to reduce the sintering temperature of nano-Ag is to apply a certain pressure during the sintering process of nano-Ag to offset the driving force reduced after lowering the sintering temperature, but this makes the process procedure cumbersome, on the other hand, it also increases the risk of device damage. Another method is to reduce the particle size of nano-Ag particles to increase the surface energy to increase the driving force for sintering, thereby reducing the sintering temperature, but this increases the production cost of nano-Ag paste and is not conducive to large-scale industrial applications [18].

Wu et al. [19] used a self-designed dispersion step to successfully incorporate various contents of Al_2O_3 nanoparticles into the solder paste in order to enhance the performance of SAC solder. After comprehensive research on the microstructure and properties of the new nanocomposite solder, some satisfactory improved results can be obtained. For example, doping with trace amounts of Al_2O_3 nanoparticles can greatly improve the wettability of solder. In addition, the joints welded with SAC–0.12Al_2O_3 have a typical ductile fracture failure mode, showing the highest shear force of 57.1 N.

Long et al. [20] conducted nanoindentation experiments on sintered silver nanoparticles, which are one of the advanced chip connection materials for high-power electronic devices that work in harsh environments. In order to further stabilize and improve the thermal conductivity, before the sintering process, SiC particles with different weight contents are mixed into the nano-Ag paste. Through the usage of the material microstructure observed by scanning electron microscope, the influence of SiC content on the mechanical properties of sintered nano-Ag paste was discussed.

Wen et al. [21] developed an effective method by adding a small amount of nano-structured polyaniline (PANI) while reducing the resistivity and improving the mechanical stability of the silver resin-based conductor. A simple chemical oxidative polymerization method was used to synthesize PANI nanomaterials for improving the properties of conductive composites, and they were characterized, and a significant improvement in electrical properties was observed.

In recent years, transient liquid phase sintering technology has attracted everyone's attention [22]. The advantages of transient liquid phase sintering include lowering the sintering temperature, accelerating the particle transport rate, and improving densification [23]. In transient liquid phase sintering, the low melting point metal melts and forms a liquid phase that diffuses into the base metal that is still in the solid state. After full reaction, the IMC is formed. The melting point of the IMC is higher than that of the low melting point metal. The phase wets the surrounding matrix metal, and the capillary force generated during the period will cause atomic rearrangement and densification, which will lead to an increase in the strength of the joint [24,25]. Transient liquid phase sintering technology includes liquid phase sintering and diffusion bonding technology [26].

Based on the transient liquid phase sintering mechanism, nano-Ag paste is used as the matrix, and the cost-effective Sn is used as the external metal. By doping the nano-size Sn paste into the nano-Ag paste, the uniform mixing of the two can be achieved. Pressureless sintering at a lower sintering temperature can form a sintered joint with a connection strength greater than 20 MPa [27].

4 Research significance of nano core–shell materials

Considering that Sn is easily oxidized, and the core–shell material can prevent the oxidation of Sn, while ensuring the uniform distribution of Sn, the performance of the nano-Ag paste is improved by the method of forming the core–shell structure. The preparation methods of metal core–shell materials generally include: solid, gas, and liquid methods.

Shang et al. [28] reflow soldered a solder ball with an initial diameter of 1.4 mm to a 523.15 K Cu substrate, and the proportion of Cu@Ag core–shell nanoparticles doped in the flux was 0–2 wt%. Then, the solder is air-cooled to room temperature. In the case of reducing the base height of the solder and increasing the diameter of the solder, the use of the core–shell structure results in an overall increase in the spreading rate of the solder.

Ji et al. [29] successfully synthesized nano Cu@Ag pastes and successfully used them to connect Cu/Cu@Ag/Cu in the air through ultrasonic-assisted sintering at a temperature as low as 160°C. The best shear strength of their sintered joints has reached more than 50 MPa. Therefore, nano Cu@Ag paste has great potential to be applied to high-temperature power device packaging.

Cao's team [30] synthesized nano-copper powder by polyol method, then mixed $SnCl_2$ and thiourea separately, and then heated to a certain temperature to form a chemical reaction. The result showed that a layer of Sn particles was wrapped on the surface of nano-copper powder. It is determined through experiments that the size of the prepared nano core–shell material is about 30 nm, and the thickness of the Sn shell is about 3 nm. The general reaction process is shown in Figure 6.

Kong et al. [31] used a heterogeneous flocculation method to coat boehmite AlOOH on zirconia tetragonal zirconium polycrystal (TZP). After sintering the mixed particles, the boehmite was converted into alumina. The TZP powder coated with Al_2O_3 is compounded into the hydroxyapatite (HAP), which greatly inhibits the formation of tricalcium phosphate (TCP) between HAP and TZP, and significantly improves the mechanical properties of the material. The author chooses that the pH value is around 7.5, the ZrO_2 particles are negatively charged, and the AlOOH particles are positively charged, so the surface of the ZrO_2 particles can be coated with AlOOH.

According to the abovementioned literature, in the preparation technology of nano core–shell materials,

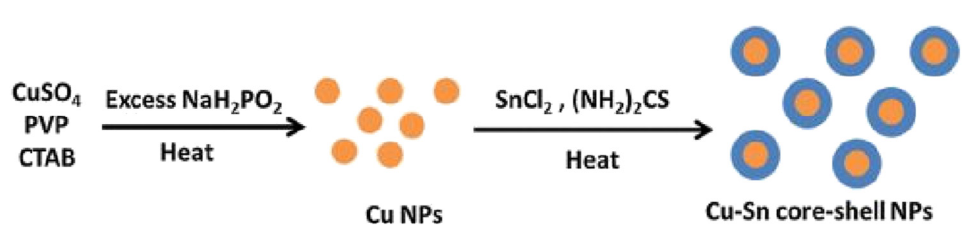

Figure 6: Preparation process of tin-coated copper nano core–shell particles.

the solid-phase method and the gas-phase reaction method have their own limitations and are rarely used. However, the liquid-phase reduction method can quickly prepare powders of the same size and the same composition by a chemical reaction method. The reaction process does not have too high requirements on the experimental device, the process is simple, and the reaction process is basically gentle. Liquid-phase reduction methods generally are of two types: co-reduction methods for preparing nano core–shell powder at one time and continuous reduction methods for nucleating first and then coating the shell [32]. The co-reduction method is limited by the properties of the metal itself, so its application range is relatively small. Heterogeneous flocculation method refers to the fact that particles with different charges attract each other and agglomerate. It is a continuous reduction method for preparing core–shell materials. This method has the advantages of mild reaction conditions and less equipment investment. Therefore, the heterogeneous flocculation method is selected for the preparation of silver-coated tin (Sn@Ag) core–shell nanoparticles [33].

5 Reliability research method of solder joints

Because the micro-bump flip chip is a high-density and high-power device, and it is in a high-temperature environment for a long time, it is necessary to study the high-temperature reliability of solder bumps. Analysis of whether the solder joints are reliable can be carried out in the following ways:

5.1 Experimental test method

There are multiple causes and forms of solder joint failure in electronic packaging, such as short circuits, solder joints, and bridging. These conditions can basically be detected by infrared analysis, metallographic section, X-ray, and other methods.

Tan et al. [34] investigated the evolution of the microstructure and shear strength of nano-Ag-sintered joints after thermal aging at 125–350°C for up to 500 h. Zhang et al. [35] evaluated the long-term reliability of nano-Ag paste sintered SiC devices by a high temperature storage process in air and vacuum at 350°C, respectively.

5.2 Theoretical method

In addition to the experimental test method, there are also theoretical methods for the failure analysis of solder joints. There are many theoretical analysis methods, but the finite element method is commonly used at present. There are many finite element software on the market, such as ABAQUS, ANSYS, etc. It is better to use finite element simulation technology to analyze the failure of solder joints. And this can save a lot of resources, time, and shorten the product development cycle.

Su et al. [36] provided an efficient and reliable method to simulate the mechanical and failure behavior of sintered nano-Ag pastes with random microporous structure.

Yang [37] simulated the chip bonding through the finite element software ANSYS, and the nano-Ag solder joints were modeled by the Anand viscoplastic constitutive model, which could reasonably simulate the stress and strain of the solder joints under thermal cyclic loading. The results show that the area where the maximum stress and strain occurs in the solder joint is in the contact area between the solder joint and the copper pillar.

6 Summary of the research in this article

(1) First, the nano-Ag paste was prepared by experiments, and the influencing factors of the strength of nano-Ag joints were analyzed. Experiments were carried out under various process parameters, and the influence of various factors on the joint strength was calculated. It was found that when the sintering temperature, pressure, time, and shear rate were increased, the shear strength of the joint could be improved to different degrees.
(2) Establish an ANSYS nano-Ag solder joint and chip bonding model. After five thermal cycle loads, the weakest link of the solder joint is the contact part between the solder joint and the copper column, which is where fatigue fractures are most likely to occur.
(3) A nano-Ag-coated Sn paste was prepared by heterogeneous flocculation method. The joint sintering performance of this Sn@Ag paste was improved, and the oxidation resistance of nano-Sn was also improved. The sintering strength of Sn@Ag paste increases with the increase in Sn content. When the Sn content reaches 5%, the shear strength of the joint reaches

the highest value of 50 MPa, which is more than 10 MPa higher than that of pure nano-Ag paste sintered joint.

(4) The nano-Ag-Sn solder joint is modeled by the method of Anand unified viscoplastic constitutive model and elastic model, and the maximum displacement of the flip chip is determined to appear at the center solder joint through finite element simulation. Through the prediction of finite element method and EPRI estimation method, it is found that the fatigue life of nano-Ag Sn solder joints is significantly higher than that of nano-Ag solder joints, indicating that adding a certain amount of tin element to nano-Ag paste can improve the reliability of solder joints.

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Data availability statement: The data of this study are available from the corresponding author upon reasonable request.

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