Article

The Realization of ZnO Nanowires Interconnection through Femtosecond Laser Irradiation of Ag Nanoparticles Solder

Yanchao Guan ^{1,2}, Genwang Wang ^{1,2}, Shouxin Zhao ³, Lianfu Wang ^{1,2}, Ye Ding ^{1,2,*} and Lijun Yang ^{1,2,*}

¹ Key Laboratory of Micro-Systems and Micro-Structures Manufacturing, Ministry of Education, Harbin Institute of Technology, Harbin 150001, China; guanyanchao@163.com (Y.G.); niskier@163.com (G.W.); wanglianfu@hit.edu.cn (L.W.)

² School of Mechatronics Engineering, Harbin Institute of Technology, Harbin 150001, China

³ School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China; zhaoshouxin@hit.edu.cn

* Correspondence: dy1992hit@hit.edu.cn (Y.D.); yljtj@hit.edu.cn (L.Y.)

Abstract: Nanowire interconnection is the basis for the construction and integration of micro-nano functional devices. But so far, it is still difficult to achieve a reliable interconnection of metal oxide nanowires. This letter proposes an approach for soldering ZnO nanowires through femtosecond laser irradiation of Ag nanoparticles solder. In this paper, the effect of femtosecond laser fluence and irradiation time on the morphology of Ag solders and the interconnection state of ZnO nanowires are studied, respectively. The I-V electrical characterization of nanowire interconnection before and after soldering is completed. The results demonstrate that ZnO nanowires achieve better interconnection. The UV light response of the ZnO-Ag-ZnO interconnection structure after soldering is investigated. The approach confirms the effectiveness of a femtosecond irradiated metal nanoparticles solder to achieve metal oxide interconnection, offering the prospect of more metal oxide nanowires interconnection and device development.

Keywords: ZnO nanowires interconnection; femtosecond laser irradiation; Ag nanoparticles solder; photoelectric property

Citation: Guan, Y.; Wang, G.; Zhao, S.; Wang, L.; Ding, Y.; Yang, L. The Realization of ZnO Nanowires Interconnection through Femtosecond Laser Irradiation of Ag Nanoparticles Solder. *Appl. Sci.* **2022**, *12*, 1004. <https://doi.org/10.3390/app12031004>

Academic Editor: Mark J. Jackson

Received: 17 December 2021

Accepted: 18 January 2022

Published: 19 January 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

In recent years, the integration density of electric devices has risen rapidly, and device sizes have gradually developed to the nanoscale [1]. As bridges of device integration, nanowires have gained extensive research and development [2]. Nanowires, especially metal oxide nanowires, have been used in a variety of applications due to their excellent electrical, optical, magnetic, and biological properties, such as in micro-nano functional devices [3], sensors [4], and batteries [5]. The effective interconnection of nanowires is inextricably linked to effective device integration. Therefore, achieving the interconnection of nanowires with high quality and low damage has become a vital issue in the manufacturing process of micro-nano devices.

In order to obtain better nanowires interconnection, scholars have proposed a variety of approaches to obtain nanowire interconnection, such as joule melt connection [6], electron beam/ion beam connection [7,8], brazing [9], and cold welding [10]. Laser welding nanowires technology has developed rapidly in the past few years [11–15]. Different from conventional laser (continuous laser, nanosecond laser, etc.)-induced “thermal processing” methods, the ultrafast laser, especially femtosecond laser welding, has a very high pulse energy density, allowing nano-welding at a low average laser power. It reduces damage to the nanowires and substrate [16], thus offering great manufacturing advantages in the field of nano-welding [17]. Its main mechanism is to induce plasmon photothermal effects on metal nanomaterials, thereby realizing the effective connection of the metal-metal and metal-metal oxide nanowires. However, laser welding of metal oxide nanowires is limited.

The absence of a plasmonic excitation photothermal effect and relative high point result in significant obstacles for the interconnection of metal oxide nanowires. To solve this issue, Li [18] used a high-energy focused laser beam to irradiate the ZnO nanowires, achieving a hot-melt connection. However, the joint melt size was large, and laser irradiation caused obvious excess ablation to the substrate after welding. Xing [19] trapped the electron-hole plasma energy by lattice defects based on the femtosecond laser two-photon absorption to realize the interconnection of the ZnO nanowires. However, this approach is only feasible for wide bandgap nanowires and cannot work well with other metal oxide nanowires.

So far, there has been no suitable laser welding method to achieve metal nanowire interconnection. This paper proposes a method to interconnect metal oxide nanowires by melting metal nanoparticles solder using a laser-induced plasmonic excitonic photothermal effect. This paper investigates the effect of femtosecond laser fluence on the melting morphology of Ag solders and the irradiation time on nanowire soldering interconnections. The electrical properties of nanowires are characterized before and after soldering. The photoelectric property of the ZnO-Ag-ZnO interconnect structure under UV light is tested.

2. Materials and Methods

The metal oxide nanowires selected in this article are ZnO nanowires (50–120 nm in diameter, 2–20 μm in length), and the solder are nanoclusters composed of multiple Ag nanoparticles (60–150 nm in diameter). They are all purchased from Jiangsu Xianfeng Nano Material Technology Co., Ltd, Nanjing, China. A solution of ZnO nanowires with a mass fraction of 0.065 g/L and a solution of Ag nanoparticles with a mass fraction of 0.125 g/L are mixed, configured into a suspended ethanol solution. The solution is spin-coated on a silicon oxide substrate with marks after being ultrasonically dispersed. The deposited silicon oxide substrate is placed on a nanomanipulation platform (MM3A-EM, Kleindiek, Reutlingen, Germany) built in a scanning electron microscope (SEM, Zeiss Sigma300). Nanoprobes (T-4-5B, GGB) are used to assemble nanowires-solder interconnection structures. The detailed assembly process of this part has been described in the reference [20]. The position of the ZnO nanowires joint before soldering is marked by SEM. The schematic diagram of the femtosecond laser (Pharos, Light Conversion, Vilnius, Lithuania) irradiation system is shown in Figure 1. The wavelength of the femtosecond laser is 1030 nm. The pulse duration of the femtosecond laser is 255 fs. The 1 MHz repetition rate of incident laser is focused by using the optical lenses with the focal length of 50 mm. The diameter of laser focus is about 10 μm . The adjustable output power of 10 mW–100 mW is used to irradiate solders. The electron beam lithography system (EBL, Nanometer Pattern Generation System, Bozeman, MT, USA) can selectively expose the sample coated with a photoresist to construct the electrode structure. After the development, the gold electrode is obtained by a thermal vacuum evaporation instrument (VZZ-300, Vnano Vacuum Technology Co., Ltd., Tianjin, China). The external Keithley 4200 A Semiconductor Characterization System (SCS, Beaverton, OR, USA) is used to characterize the electrical properties of the interconnection structure by the probe station. The photoelectric response based on the interconnection structure is tested under 350 nm UV light irradiation.

Figure 1. Schematic diagram of the femtosecond laser irradiation system.

3. Results and Discussion

3.1. Femtosecond Laser Irradiates Ag Nanoparticles Solder

Figure 2 shows the SEM images of Ag solders before and after the femtosecond laser irradiation. The laser fluence are 29.25 mJ/cm², 42.25 mJ/cm², 55.25 mJ/cm², and 68.25 mJ/cm², respectively, and the irradiation time is 30 s in Figure 2a–h. Figure 2a–d shows the morphology of Ag solders before irradiation. Figure 2e,f shows that each of the nanoparticle’s edges in the nano-solder appear to be slightly melted and blurry after irradiation. When the femtosecond laser irradiates the Ag nanoparticles, plasmon oscillation is generated on the surface of the nanoparticles. The intensity of the electromagnetic field surrounding Ag nanoparticles is increased, exhibiting nonlinear absorption of incident photons. The plasmon resonance enhances the photothermal effect to produce multiple “hot spots” around the Ag nanoparticles, leading to melting and deformation of the Ag nanoparticles’ edge. However, the overall melting phenomenon in Figure 2e,f is not obvious. This is attributed to the low laser fluence and the weak plasmon photothermal effect between the nanoparticles. In Figure 2g, a large melting deformation between the nanoparticles is apparent, and the blurring phenomenon of the Ag particles edges has vanished. This results from the higher fluidity of the locally molten metal material under the higher laser fluence. The gap between the nanoparticles is filled gradually. The flowing molten solder acts as a bridge to connect the individual nanoparticles, thus achieving good bonding. In Figure 2h, the nanoparticles exhibit significant melting deformation as well as even overall fracture and shrinkage, which can be explained by the high laser fluence and the significant photothermal effect of plasma excitation resonance. The large melt deformation of the nanoparticles may cause the displacement of solders and poor nanowire-solder contact during the soldering process, which is detrimental to nanowire soldering. Through the comparison of melting morphologies of Ag nanoparticles solders, optimized melting of Ag solders can be obtained under a laser fluence of 55.25 mJ/cm².

Figure 2. SEM images of Ag nano-solder before and after femtosecond laser irradiation with different fluence: (a,e): 29.25 mJ/cm²; (b,f): 42.25 mJ/cm²; (c,g): 55.25 mJ/cm²; (d,h): 68.25 mJ/cm².

3.2. Soldering Interconnection of the ZnO Nanowires and Ag Solder

Based on the experimental results in Section 3.1, the laser fluence of 55.25 mJ/cm² and laser irradiation time of 30 s is used to interconnect ZnO nanowires. The results are demonstrated in Figure 3. The nano-solder is accurately positioned at the joint of the nanowire using the nanomanipulation platform based on the SEM, as shown in Figure 3b. Due to the high melting point, Figure 3c shows ZnO nanowires undergo no morphological changes after femtosecond laser irradiation, which proves that the nanowires have not been damaged to a large extent. In contrast, Ag nano-solder undergoes a significant morphological change. The nanoparticles are transformed from individual stand-alone to sticky as a whole and bond well, indicating that the Ag nano-solder has melted sufficiently. Meanwhile, the Ag nano-solder is found to be attached to the nanowires with the absence of position change. This phenomenon ensures good soldering interconnection of the ZnO nanowires and Ag nano-solder.

Figure 3. SEM images of the interconnection melting state at 55.25 mJ/cm². (a,c) are SEM images of ZnO nanowires-Ag solder interconnection after irradiation; (b) SEM images of ZnO nanowires and Ag solder interconnection before irradiation.

To better explore the interconnection between nanowires and nano-solder, Figure 4 shows the SEM image of longer irradiation time of 45 s, 60 s, and 75 s, respectively. Figure 4a–c shows the morphology of the interconnection joint before irradiation. Figure 4 shows that the Ag solders have all melted to a greater degree, and the morphological change of the nanoparticles becomes more evident as the irradiation time increases. As shown in Figure 4d,e, the better soldering interconnections have been realized. However, Figure 4f

shows that the ZnO nanowires are broken after 75 s of femtosecond laser irradiation. The analysis goes that the plasmon effect between the Ag nanoparticles is stronger under the long-term irradiation of the femtosecond laser, resulting in the transmission of a considerable amount of heat to the nanowires through Ag nanoparticles. The nanowires appear to be ablated and cracked after being heated. The phenomenon also indicates that, even if the ZnO nanowire's melting point is high, the photothermal effect is sufficient to destroy the nanowires when exposed to a long-term irradiation time. By comparing the melting state of the Ag solder at different irradiation times, it can be discovered that the melting of Figure 4e is more pronounced, and the Ag solder achieves better melt fluidity, which is more favorable for nanowire soldering. The above experiments prove that a femtosecond laser fluence of 55.25 mJ/cm^2 and an irradiation time of 60 s are more effective for soldering interconnection of ZnO nanowires.

Figure 4. SEM images of the interconnection melting state at different irradiation time before and after soldering: (a,d) 45 s; (b,e) 60 s; (c,f) 75 s.

3.3. Electrical Characterizations of ZnO Nanowires and Ag Nano-Solder Interconnection

In order to explore the melting state of the interface between the nanowires and nano-solder, the typical characterization method is the transmission electron microscope (TEM). However, TEM characterization is rather challenging due to the large size of heterojunctions composed of the nano-solder and nanowires. Hence, this paper tested the electrical properties before and after femtosecond laser irradiation to determine whether good soldering was achieved or not. The femtosecond laser fluence of 55.25 mJ/cm^2 and irradiation time of 60 s are more beneficial for nanowires interconnection (according to the experimental results in Section 3.2). Thus, the structure shown in Figure 5a is used to characterize the electrical properties of nanowires interconnection under the above femtosecond laser irradiation conditions. Two Au electrodes are deposited on both ends of the interconnected nanowires. Voltage-current scanning is from -6 V to 6 V , and the acquisition time of each point is 1 s. Figure 5b shows the current conduction of the nanowire interconnection structure before and after femtosecond laser soldering. The experiment is established in a daylight environment. It can be seen that the conduction current of the nanowires is feeble before soldering, and there is almost no conduction under positive pressure. After 60 s of femtosecond laser irradiation, the ZnO nanowires have successfully achieved current conduction. Under positive bias, the interconnection current reached a maximum of $0.7 \mu\text{A}$. At the same time, the I-V curve shows the typical I-V response of the dual Schottky barrier, which is due to the contact barrier between the Au electrodes and the ZnO nanowires. There is no electron energy barrier at the interface since the electron affinity of ZnO is 4.5 eV and the work function of Ag is 4.26 eV . Besides, Au has a work function of 5.1 eV . There is an electron energy barrier at the contact since the work function of Au is massively larger than the electron affinity of ZnO [21]. Figure 5b shows that the

negative bias junction current is slightly larger than the positive bias current, which may be caused by the inconsistent contact barrier between the electrode and the nanowire on both sides. In short, the results illustrate the successful soldering interconnection through electrical characterizations of ZnO nanowires and Ag nano-solder interconnection before and after femtosecond laser irradiation.

Figure 5. Electrical characterizations of ZnO nanowires and Ag nano-solder interconnection. (a) Test electrode of nanowire interconnection structure; (b) I–V electrical test curve before and after soldering.

3.4. Photoelectric Properties Test of the ZnO-Ag-ZnO Interconnection Structure

Considering that ZnO nanowires have good ultraviolet (UV) photoelectric response characteristics, we tested the photoelectric properties of the ZnO-Ag interconnection structure after soldering to further demonstrate its potential application. Figure 6a illustrates the schematic image of the UV detector based on the ZnO-Ag-ZnO interconnection structure. The I–V test results are shown in Figure 6b. It can be seen that the detection current is small when the UV light is off, and the detection resistance is around 13.5 MΩ. When the UV light is turned on, the current of both ends is significantly increased, and it increases to 1.47 μA when a bias voltage is 5 V. The detection resistance is around 3.4 MΩ, and the resistance of the structure is reduced to one-fourth of the original when the lights were off. The results demonstrate that an interconnected structure provides good photoelectric enhancement under 350 nm UV light irradiation. In order to judge the photoelectric performance of the device in a better way, Figure 6c further illustrates the current response of the interconnected ZnO-Ag device upon 350 nm UV light on-off cycles. The test is carried out with a bias voltage of 5 V at both ends. It can be discovered that the dark current is about 0.2 μA, and the photocurrent jumps to about 1.0 μA–1.5 μA. During continuous UV light on-off for 70 s, the photoresponse changes periodically, proving a good reproducibility and stability for interconnection structure. Figure 6d shows the photoelectric response curve of a single cycle from 25 s to 45 s in Figure 6a. The response time of the device to 350 nm light detection (the photocurrent increases from 10% of the peak value to 90%) is 8 s, and the recovery time (the photocurrent changes from 90% of the peak value to 10%) is 10 s. These results show that the ZnO-Ag heterogeneous interconnection structure has a good UV photoelectric response, which provides a novel method for constructing various photoelectric devices through the soldering of nanowires.

Figure 6. The photoelectric properties test of the ZnO-Ag-ZnO interconnection structure. (a) Schematic of the device based on the ZnO-Ag-ZnO interconnect structure; (b) I-V curve of photocurrent and dark current of the device; (c) photoresponse curve of the interconnection device; (d) the response and recovery time of a single response curve.

4. Conclusions

In conclusion, this paper proves an effective method to interconnect metal oxide nanowires using femtosecond laser irradiation of a metal nano-solder. The nanowire interconnection state is controlled by the laser fluence and irradiation time. The results indicate that a femtosecond laser fluence of 55.25 mJ/cm^2 and an irradiation time of 60 s are favorable for nanowire soldering. It was found that the current increased significantly after soldering through the electrical test, confirming that the ZnO nanowires and Ag solder successfully achieve a better interconnection. Under UV light irradiation, the resistance was reduced to approximately one-quarter of the resistance without light. The device's response time to 350 nm UV light detection was 8 s, and its recovery time was 10 s, showing its potential as a UV detector. The effect of femtosecond lasers with different laser wavelengths and pulse duration on nanowires interconnection requires further exploration. It is expected to interconnect more difficult-to-melt metal oxide nanowires by soldering and develop more metal oxide nanowire functional devices in future.

Author Contributions: Conceptualization, Y.G.; methodology, Y.G., G.W. and L.W.; validation, S.Z., Y.D. and L.Y.; formal analysis, Y.G.; investigation, Y.G. and Y.D.; resources, Y.D. and L.Y.; data curation, Y.G.; writing—original draft preparation, Y.G.; writing—review and editing, Y.D. and L.Y.; supervision, L.Y.; project administration, Y.D. and L.Y.; funding acquisition, L.Y. and Y.D. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Natural Science Foundation of China, Grant number 52105437.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: Thanks to Bai Cheng for offering valuable guidance during the writing of this manuscript. Thanks to Shang Huiming for his help on the experiment.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Cao, Q.; Tersoff, J.; Farmer, D.B. Carbon nanotube transistors scaled to a 40-nanometer footprint. *Science* **2017**, *356*, 1369–1372. [\[CrossRef\]](#)
2. Li, X.; Gao, F.; Gu, Z. Nanowire joining methods. *Open. Surf. Sci. J.* **2011**, *3*, 91–104. [\[CrossRef\]](#)
3. Xu, S.; Qin, Y.; Xu, C.; Wei, Y.; Yang, R.; Wang, Z.L. Self-powered nanowire devices. *Nat. Nanotechnol.* **2010**, *5*, 366–373. [\[CrossRef\]](#) [\[PubMed\]](#)
4. Liu, X.; Gu, L.; Zhang, Q.; Wu, J.; Long, Y.; Fan, Z. All-printable band-edge modulated ZnO nanowire photodetectors with ultra-high detectivity. *Nat. Commun.* **2014**, *5*, 1–9. [\[CrossRef\]](#) [\[PubMed\]](#)
5. Cheng, Y.W.; Chen, C.H.; Yang, S.W.; Li, Y.C.; Peng, B.L.; Chang, C.C.; Wang, R.C.; Liu, C.P. Freestanding three-dimensional CuO/NiO Core–Shell nanowire arrays as high-performance lithium-ion battery anode. *Sci. Rep.* **2018**, *8*, 1–10. [\[CrossRef\]](#) [\[PubMed\]](#)
6. Tohmyoh, H.; Imaizumi, T.; Hayashi, H.; Saka, M. Welding of Pt nanowires by Joule heating. *Scripta Mater.* **2007**, *57*, 953–956. [\[CrossRef\]](#)
7. Shen, D.; Chen, D.; Yang, Z.; Liu, H.; Chen, T.; Sun, L.; Fukuda, T. Electron beam irradiation induced multiwalled carbon nanotubes fusion inside SEM. *Scanning* **2017**, *2017*, 8563931. [\[CrossRef\]](#) [\[PubMed\]](#)
8. Wang, Z.; Yu, L.; Zhang, W.; Ding, Y.; Li, Y.; Han, J.; Zhu, Z.Y.; Xu, H.J.; He, G.W.; Chen, Y.; et al. Amorphous molecular junctions produced by ion irradiation on carbon nanotubes. *Phys. Lett. A* **2004**, *324*, 321–325. [\[CrossRef\]](#)
9. Peng, Y.; Cullis, T.; Inkson, B. Bottom-up Nanoconstruction by the welding of individual metallic nanoobjects using nanoscale solder. *Nano Lett.* **2009**, *9*, 91–96. [\[CrossRef\]](#)
10. Lu, Y.; Huang, J.Y.; Wang, C.; Sun, S.H.; Lou, J. Cold welding of ultrathin gold nanowires. *Nat. Nanotechnol.* **2010**, *5*, 218–224. [\[CrossRef\]](#)
11. Lin, L.C.; Xing, S.L.; Huo, J.P.; Xiao, Y.; Peng, P.; Shen, D.Z.; Liu, L.; Zou, G.S. Research Progress of Ultrafast Laser-Induced Nanowires Joining Technology. *Chin. J. Lasers* **2021**, *48*, 9–25.
12. Lin, L.C.; Liu, L.; Peng, P.; Zou, G.; Duley, W.W. In situ nanojoining of Y-and T-shaped silver nanowires structures using femtosecond laser radiation. *Nanotechnology* **2016**, *27*, 125201. [\[CrossRef\]](#)
13. Li, Q.; Liu, G.; Yang, H.; Wang, W.; Luo, S.; Dai, S.; Qiu, M. Optically controlled local nanosoldering of metal nanowires. *Appl. Phys. Lett.* **2016**, *108*, 193101. [\[CrossRef\]](#)
14. Feng, J.; Tian, Y.; Wang, S.; Xiao, M.; Hui, Z.; Hang, C.; Duley, W.W.; Zhou, Y.N. Femtosecond laser irradiation induced heterojunctions between carbon nanofibers and silver nanowires for a flexible strain sensor. *J. Mater. Sci. Technol.* **2021**, *84*, 139–146. [\[CrossRef\]](#)
15. Dai, S.; Li, Q.; Liu, G.; Yang, H.; Yang, Y.; Zhao, D.; Wei, W.; Qiu, M. Laser-induced single point nanowelding of silver nanowires. *Appl. Phys. Lett.* **2016**, *108*, 121103. [\[CrossRef\]](#)
16. Ghosh, P.; Lu, J.; Chen, Z.; Yang, H.; Qiu, M.; Li, Q. Photothermal-Induced Nanowelding of Metal–Semiconductor Heterojunction in Integrated Nanowire Units. *Adv. Electron. Mater.* **2018**, *4*, 1700614. [\[CrossRef\]](#)
17. Zou, G.S.; Lin, L.C.; Deng, Z.Y.; Jia, Q.; Feng, B.; Wang, W.A.; Xing, S.L. Ultrafast laser nanojoining and its applications in the manufacturing of micro-nano devices. *Chin. J. Lasers* **2021**, *48*, 1502001.
18. Liu, G.; Li, Q.; Qiu, M. Sacrificial solder based nanowelding of ZnO nanowires. *J. Phys. Conf. Ser.* **2016**, *680*, 012027. [\[CrossRef\]](#)
19. Xing, S.; Lin, L.; Zou, G.; Duley, W.W.; Liu, L.; Zhou, Y.N. Two-photon absorption induced nanowelding for assembling ZnO nanowires with enhanced photoelectrical properties. *Appl. Phys. Lett.* **2019**, *115*, 103101. [\[CrossRef\]](#)
20. Guan, Y.; Wang, G.; Wang, Y.; Ding, Y.; Yang, L. Assembly and Braze of Nanowires Interconnection Structure Based on Nanomanipulation. *Chin. J. Lasers* **2021**, *48*, 287–295.
21. Choi, J.; You, X.; Kim, C.; Park, J.; Pak, J.J. Power generating characteristics of zinc oxide nanorods grown on a flexible substrate by a hydrothermal method. *J. Elector. Eng. Technol.* **2010**, *5*, 640–645. [\[CrossRef\]](#)