



A Crystal Growth Method for Water

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Water is crystallized into ice using a simple setup and $\Phi 12$ mm \times 16 mm ice have been achieved. It proves the feasibility of a method to grow crystal. This method is very similar with the temperature gradient technique. But the crystal growth direction is verse with the temperature gradient technique. This method can grow crystal from the top to the bottom of the crucible. The key point of this method is the special crucible shape. Some of the potential advantages and disadvantages of this method are discussed.

Key Words: Crystal growth, Temperature gradient technique, Direction.

INTRODUCTION

The crystal growth direction is very important. The crystal growth direction of the vertical Bridgman method is parallel to the gravitation direction but verse with the gravitation direction. The crystal growth direction of the horizontal Bridgman method is vertical to the gravitation direction. So there are many differences between the vertical Bridgman method and the horizontal Bridgman method on the convection of the liquid-solid interface, the shape of the liquid-solid interface, the crucible requirement, etc. So the crystals grown by those different methods are different.

The temperature gradient technique (TGT) is a well known method to grow crystal. The temperature gradient technique has so many advantages for crystal growth¹⁻⁸. For example, the temperature gradient technique can grow the crystals with melting point higher than 2000 °C. The temperature gradient technique doesn't need the mechanical equipments. Many commercialized crystals have been successfully grown by the temperature gradient technique, such as the Al₂O₃, yttrium aluminium garnet (YAG), etc. The crystal growth direction of the temperature gradient technique is parallel to the gravity direction but verse with the gravity direction. It means that temperature gradient technique grows the crystals from the bottom of the crucible to the top of the crucible. In this paper, water is solidified using a simple setup and *ca.* Φ 12 mm \times 16 mm ice have been achieved. It proves the feasibility of a method to grow crystal. This method is similar with the temperature gradient technique. But the crystal growth direction is verse with the temperature gradient technique. This method can grow crystal from the top to the bottom of the crucible. The key

point to achieve such crystal growth direction is the special crucible shape. The crucible is a connecting vessel. It makes this special crystal growth direction feasible. Some of the potential advantages and disadvantages of this method are discussed.

EXPERIMENTAL

Crucible design: The crucible is shown in the Fig. 1. This crucible is made of quartz glasses. The length of the left side of the crucible is 90 mm. The length of the right side of the crucible is 160 mm. It can be seen that this crucible is a connecting vessel. The right side of this connecting vessel is open. And the material can be put into the crucible from the right side. The left side is used to grow crystal. The inner diameter of the left side of the crucible is becoming more and more smaller from the top (14 mm) to the bottom (10 mm).

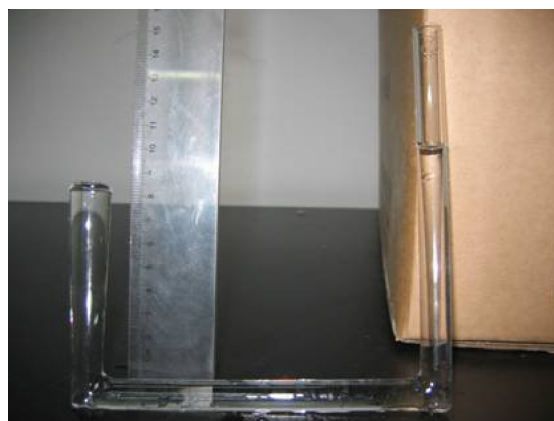


Fig. 1. Crucible

Water crystallization: A simple setup is shown in the Fig. 2. And water is solidified into ice using this setup. The crucible is put in a big container. The container is full of the mixture of water and ice. The top of the crucible left side is 20 mm higher than the surface of the mixture of water and ice. Water is poured into the crucible from the right side until the horizontal surface of the water in the right side of the crucible is a little higher than that of the crucible at the left side. The crucible of the left side is connected with an iron ingot.



Fig. 2. Setup for the water crystallization

The liquid nitrogen is slowly and continuously poured onto the iron ingot. The flow velocity of the liquid nitrogen is about 30 mL/min. The room temperature is 5 °C. After 0.5 h, *ca.* 16 mm long ice have been achieved at the left side of the crucible (Fig. 3).



Fig. 3. 16 mm long ice achieved in the left side of the crucible

RESULTS AND DISCUSSION

It's obviously that the setup in the Fig. 2 is very simple. But water have successfully been crystallized into the ice from the top of the crucible. It is the first time that the crystal has been achieved from the top of the crucible to the bottom of the crucible along with the gravitation direction. This proves the feasibility of a crystal growth method.

A furnace is designed and being built to develop this new crystal growth method now. The schematic diagram of this furnace is shown in the Fig. 4. The shape of the crucible in the Fig. 4 is same with the shape of the crucible in the Fig. 1. The crucible's a connecting vessel. It's the key point to grow crystal along the gravitation direction. First, if the liquid surface of the right side of the crucible is kept higher than the liquid surface of the left side of the crucible, then the left side of the crucible can continuously get the liquid phase. The detachment of solid phase and liquid phase will not happen. Second, the inner diameter of the crucible is becoming more and more smaller from the top to the bottom. The solid phase will not fall down into the liquid phase. The furnace in the Fig. 4 can heat the crucible and the materials. Thus the crystals with high melting point could be grown. The heating system is composed of the temperature controlling equipment, heating elements and the temperature monitor thermocouple. The furnace in the Fig. 4 can also cool down the crucible and the materials by the metal cooling rod. The materials are first put in the crucible. Then the crucible is put in the furnace. The furnace will be heated till the materials in the crucible are melted. Then the cooling water begins to flow through the metal cooling rod and crystal be achieved. Though the setup in the Fig. 2 has no heating system, the crystallization principle of it is similar with the furnace in the Fig. 4.

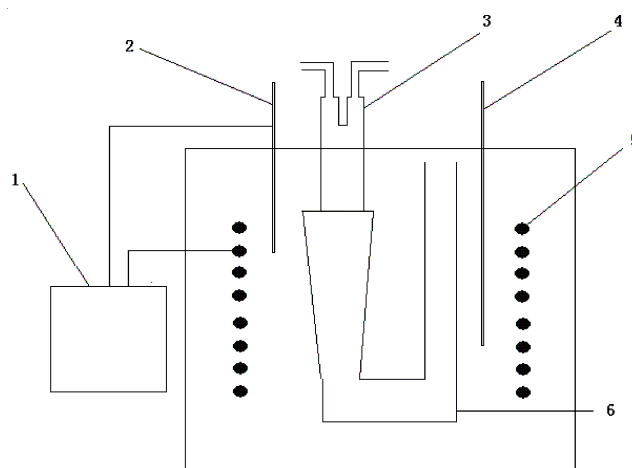


Fig. 4. Schematic of the furnace. 1. Temperature controlling equipment. 2. Temperature monitor thermocouple. 3. Metal cooling rod. 4. Temperature monitor thermocouple. 5. Heating elements. 6. Crucible

The setup in the Fig. 2 and the furnace in the Fig. 4 are demonstrating a crystal growth method. This method is feasible to grow crystal along the gravitation direction. This method is very similar with the temperature gradient technique. But the crystal growth direction of this method is reverse with the temperature gradient technique.

This method should have some advantages compared with the temperature gradient technique. For example, this method is expected to be more easier to get rid of the bubbles. For the temperature gradient technique and the vertical Bridgman method, even the liquid-solid crystal growth interface is convex, the bubbles will also be trapped by the interface. Because the middle of the interface is higher than the brim of the interface. And the bubbles are always flowing upward. But for this new method, if the liquid-solid crystal growth interface is convex, then the brim of the interface is higher than the middle of the interface. Therefore the bubbles will tend to flow to the brim and thus difficult to be trapped by the interface. It means the crystals grown by this method should have less bubbles. Considering the bubble is one kind of the main crystal defects, this method has one obvious advantage.

It is obvious that this method will be different from the other traditional methods on many things, such as the convection of the melt, the liquid-solid interface stability, *etc.* The advantages of this method are far from being fully understood. The key point of this method is the special crucible design. If the liquid surface of the right side crucible is being kept slightly little higher than the liquid surface of the left side crucible during the whole crystal growth process, then the convection status of the liquid-solid crystal growth interface will be very like the Czochralski method. But the solidified crystal has no direct contact with the air for this method. So the crystals achieved by this method are expected to be better than the crystals achieved by the Czochralski method. The disadvantages of this method are also obvious, the difficult crucible machining and the materials handling process, no good ways

to control the temperature gradient for the moment, *etc.* But some of these disadvantages should be overcome with the development of this method.

Conclusion

Water is crystallized into ice using a simple setup. The crystal growth direction is from the top of the crucible to the bottom of the crucible. This results demonstrate the feasibility of a crystal growth method. The key point to of this method is the special crucible design. The crucible is a connecting vessel. Some of the potential advantages and disadvantages of this crystal method are discussed.

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REFERENCES

1. X.H. Zeng, G.J. Zhao, J. Xu, H.J. Li, X.M. He, H.Y. Pang and M.Y. Jie, *J. Cryst. Growth*, **274**, 495 (2005).
2. H.J. Li, J. Xu, L.B. Su, B.X. Jiang, G.J. Zhao, G.Q. Zhou and Y.J. Dong, *Cryst. Res. Technol.*, **42**, 107 (2007).
3. G.J. Zhao, X.H. Zeng, J. Xu and Y.Z. Zhou, *Phys. Status Solidi(a)*, **199**, 355 (2003).
4. J.W. Xu, Y.Z. Zhou, G.Q. Zhou, K. Xu, P.Z. Deng and J. Xu, *J. Synth. Cryst.*, **27**, 50 (1998).
5. Y.Z. Zhou, *J. Cryst. Growth*, **78**, 31 (1986).
6. Y.Z. Zhou, J. Xu, J.L. Shi and G.J. Zhao, Patent 2745959, Shanghai, China (2005).
7. J. Xu, X.D. Chen and W. Chen, Patent ZL99124262.9, Shanghai, China (2002).
8. Y.Z. Zhou, Patent 85100534.9, Shanghai, China (1988).