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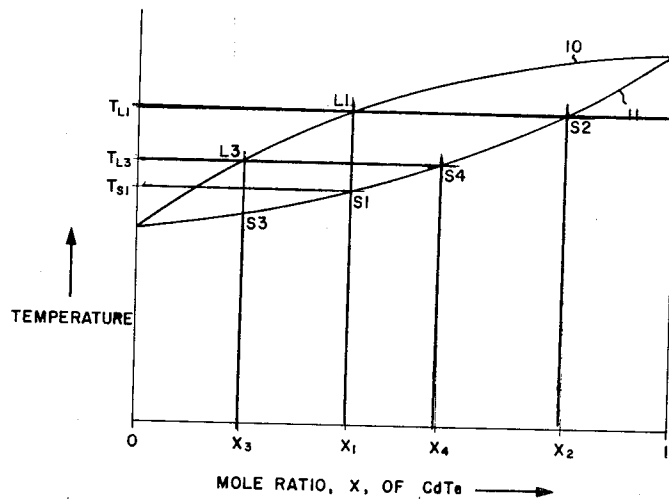
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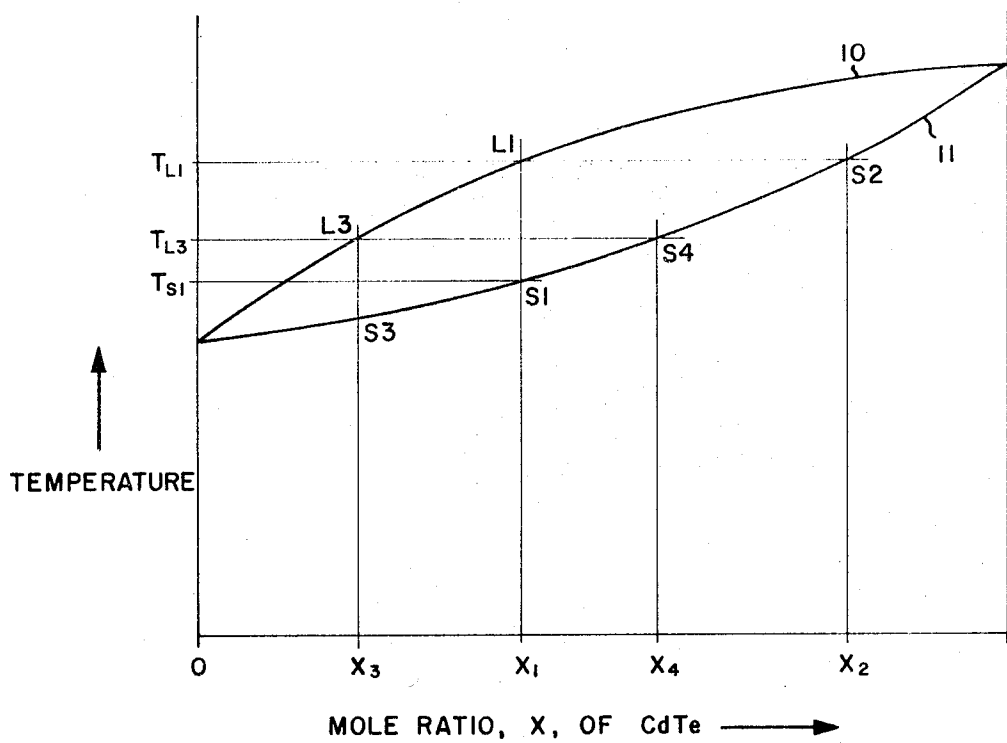
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[54] **METHOD FOR REDUCING COMPOSITIONAL GRADIENTS IN  $Hg_{1-x}Cd_xTe$**   
2 Claims, 1 Drawing Fig.

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148/1.6  
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**ABSTRACT:** Compositional gradients in a body of  $Hg_{1-x}Cd_xTe$  can be removed by annealing the body at a temperature which is greater than the solidus temperature and less than the liquidus temperature for the average composition of the body.





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## METHOD FOR REDUCING COMPOSITIONAL GRADIENTS IN $Hg_{1-x}Cd_xTe$

### BACKGROUND OF THE INVENTION

The development of solid-state detectors of wave lengths within the infrared portion of the electromagnetic spectrum has led to the use of semiconductor alloys having the proper energy gap for intrinsic photoconductivity at wave lengths within the range of 1.7 to 30 microns. One successful intrinsic detector material that has been developed for photoconductive detectors is mercury cadmium telluride, a semiconductor material which is an alloy of a semimetal, mercury telluride, and a semiconductor, cadmium telluride. The mole ratio,  $x$ , of cadmium telluride in the alloy determines the energy gap and therefore the optical and semiconducting properties of the alloy. Since the energy gap of mercury cadmium telluride is determined by the composition of the alloy, it is highly desirable to produce an ingot of mercury telluride having a uniform composition.

It has been extremely difficult to produce an ingot of  $Hg_{1-x}Cd_xTe$  having a constant composition. This difficulty arises from the tendency of the compounds to segregate when cooling, giving different compositional regions within the ingot that has been formed. The reason for this tendency to segregate can be explained graphically by a phase diagram, FIG. 1, which has as its abscissa the composition, or mole ratio, of the material and as its ordinate the temperature.

The phase diagram for  $Hg_{1-x}Cd_xTe$  shows three regions, or phases: The liquid phase, which is the region above the liquidus line 10, the solid phase, which is the region below the solidus line 11, and the liquid plus solid, or "slush" phase, the lens-shaped region between the liquidus and solidus lines. When a certain mole ratio,  $x_1$ , of CdTe within the liquid phase is cooled to a temperature  $T_{L1}$  located on the liquidus line, material having a mole ratio  $x_2$  is precipitated. This is caused by the fact that the liquidus and solidus lines are separate, and for a given temperature  $T_{L1}$  there is a point L1 on the liquidus line which corresponds to a mole ratio  $x_1$  and a point S2 on the solidus line which corresponds to a mole ratio  $x_2$ . Therefore, the first precipitated  $Hg_{1-x}Cd_xTe$  has a mole ratio  $x_2$ . As the CdTe rich material is precipitated out, the amount CdTe remaining in the liquid is depleted so as to relocate the composition of the liquid to a point at the left of point L1. Upon further cooling, the liquid having composition  $x_3$  reaches point L3 and a solid is precipitated out of composition  $x_4$ . For this reason an ingot of  $Hg_{1-x}Cd_xTe$  exhibits compositional gradients over its length.

Growth of mercury cadmium telluride can be accomplished by the use of the modified Bridgman method described by E. L. Stelzer et al. in the *IEEE Transactions on Electron Devices*, pages 880-884, Oct. 1969. In this method stoichiometric amounts of the three elements, mercury, cadmium, and tellurium, plus some excess mercury are loaded into a thick wall quartz capsule, which is then evacuated and sealed off. The sealed capsule is heated in a furnace and rocked back and forth to insure mixing, after which it is solidified from one end by sequentially cooling the three zones of the furnace. Rapid solidification reduces thermal segregation, resulting in a large single crystal ingot containing a dendritic structure, with alternating regions of high and low mole ratios. As the cooling rate is increased, the dendritic structure becomes finer. A subsequent high-temperature anneal, at a temperature below the solidus line is then used to remove the dendrites and a low temperature anneal is used to adjust stoichiometry. While it is possible to remove the dendritic microscopic compositional gradients, which are generally less than 1 mm., the high-temperature annealing step takes weeks and leaves macroscopic  $x$  gradients on the order of several millimeters unaffected. Removal of macroscopic  $x$  gradients by this method takes months or even years and is therefore impractical.

A process known as zone leveling can be used to remove macroscopic compositional gradients. Zone leveling entails heating a small region of the ingot to above the melting tem-

perature, and then advancing the molten region, or zone through the length of the ingot at a rate which is slow enough for thermal equilibrium to be approximately maintained. As the zone advances, the material left behind solidifies and new material is melted. The first material to resolidify has a greater concentration of CdTe than the material which has yet to be melted. As the zone progresses, the concentration of the liquid changes until it attains a concentration which produces, upon solidification, material which has a concentration which is equal to the concentration of the region about to be melted. When this condition is attained, the concentrations of the solids entering and leaving the zone are equal, and hence no further change of concentration occurs in the zone or in the solid freezing from it until the zone reaches the end of the ingot.

The disadvantage of the zone-leveling process is that the movement of the zone is very slow, since the ingot must be in near thermal equilibrium. An additional disadvantage is that it is difficult to control the liquid-solid interface and therefore compositional gradients may still occur.

### SUMMARY OF THE INVENTION

In the present invention, compositional gradients in an ingot of  $Hg_{1-x}Cd_xTe$  can be removed by annealing at a temperature which is greater than the solidus temperature and less than the liquidus temperature for the average composition of the ingot. Due to the greatly reduced annealing time with this method,  $Hg_{1-x}Cd_xTe$  of uniform composition can be produced by forming ingots quickly, removing the compositional gradients by annealing the body at a temperature above the solidus temperature and below the liquidus temperature for the average composition of the body, further annealing the body at a temperature near but below the solidus temperature, and adjusting stoichiometry with a low-temperature anneal.

### BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a (temperature, mole ratio) phase diagram for  $Hg_{1-x}Cd_xTe$ .

### BRIEF DESCRIPTION OF THE PREFERRED EMBODIMENT

In the present invention, an ingot  $Hg_{1-x}Cd_xTe$  having dendrites and compositional gradients is heated to a temperature above the solidus line and yet below the liquidus line for the average composition  $x_1$  of the ingot. This shifts the composition of the precipitated material within the dendritic regions closer and closer to the average composition  $x_1$ . This process occurs at a much faster rate than in a similar process employing heating below the solidus line because the material has a greater diffusibility when heated above the solidus line. The greater diffusibility is due both to the higher temperature of the annealing and also to the fact that for a given temperature  $T_{L3}$  between the solidus and liquidus temperatures  $T_{L1}$  and  $T_{S1}$  of average composition  $x_1$ , that material having a smaller concentration of CdTe than  $x_3$  is liquid, thus allowing it to flow and mix. Due to this higher diffusibility and mixing, it is possible to remove macroscopic compositional gradients as well as microscopic ones. By this method, one can obtain an ingot of  $Hg_{1-x}Cd_xTe$  which is closer to the desired uniformity over greater distances in a much shorter time than has been possible by prior art techniques.

It can be seen that the higher the temperature is within the specified range, the greater the diffusibility of the material is, and therefore annealing time is at a minimum when the temperature is just below the liquidus temperature.

In one preparation, an ingot having a composition ranging from  $x=0.130$  to  $x=0.318$  over 13 mm. was annealed for 10 days at a temperature  $T=755^\circ C.$ , which is greater than the solidus temperature and less than the liquidus temperature for the average composition,  $x=0.270$ . The resultant ingot had a composition  $x=0.270\pm 0.02$  over the same distance.

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A second ingot annealed at 750° C. for 8 days was found to have a composition  $x=0.561\pm 0.014$  across the diameter of 13 mm.

Due to the greatly reduced annealing time obtained by this method,  $Hg_{1-x}Cd_xTe$  of uniform composition which is suitable for detectors can be produced by forming ingots quickly, removing macroscopic compositional gradients by annealing the body at a temperature which is greater than the solidus temperature and less than the liquidus temperature of the average composition of the ingot, removing microscopic compositional gradients with further annealing at a temperature near but below the solidus temperature, and adjusting stoichiometry with a low-temperature anneal.

The embodiments of the invention in which an exclusive

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property or right is claimed are defined as follows:

1. A method for reducing compositional gradients in a body of  $Hg_{1-x}Cd_xTe$  wherein the body is annealed at a temperature which is greater than the solidus temperature and less than the liquidus temperature for the average composition of said body.

2. The method in accordance with claim 1 and further comprising:

annealing the body at a temperature near but below the solidus temperature for the average composition of said body, and

annealing the body at a lower temperature to adjust stoichiometry.

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