ZONE REFINING

Zone refining is a technique used to purify materials, mainly semiconductors by melting a short region (i.e., zone) and causing this liquid zone to travel slowly through a relatively long rod of the solid.

After going through this learning aid, learner will be able to
• Interpret the concept of Zone refining.
• Develop an understanding of zone refining with respect to the phase diagram.
• Classify the kinds of materials or compounds that can be purified by the process of zone refining.

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FOR many semiconductor chips and other sophisticated electronic materials, elements such as germanium and silicon are needed in their ultra-pure state so as to achieve the required properties...

DO you know how elements are obtained in such pure state?
Definitions and Keywords

• PHASE DIAGRAM- A phase diagram is a diagram that shows the phases that co-exist at equilibrium at different compositions and temperature.

• CRYSTALLIZATION- Growth of solid from a melt or a solution is defined as crystallisation.

• TEMPERATURE - (T)

• LEVER RULE– A tool which enables us to determine the relative amounts of 2 phases or the weight percentages of 2 phases in a binary phase diagram.
Above drawn is the temperature vs composition phase diagram. A tie line is first drawn from point O parallel to x axis to determine the weight percentages in solid and liquid form for point O. Expressions to determine solid and liquid weight percentages are

- Weight percentage of solid phase = \( \frac{w_o - W_l}{W_s - W_l} \)
- Weight percentage of liquid phase = \( \frac{W_s - w_o}{W_s - W_l} \)
Explanation of ZONE REFINING Technique

Zone refining technique is used to purify an element or compound or for the formation of a single crystal by melting a short region (i.e., zone) and causing this liquid zone to travel slowly through a relatively long ingot, or charge, of the solid.

This process is based on a very important fact that the solid to crystallise first from the melt is purer in A or the element to be obtained than the liquid.

Here, what we apply is the Lever Rule for the basic understanding of concept involved in Zone Refining.
As shown in the above diagram, it is an enlarged view of binary phase diagram of 2 components, A and B, let A be the element to be obtained and B be the impurity. The 2 lines are liquidous and solidous lines and the 3 regions are liquid on the top, solid on the bottom and liquid+ solid phase in between the 2 lines.

Lets consider solidification of the material at initial composition Cs. Thus solidification starts at temperature (T 1) and just below this temperature when it starts to solidify, a small quantity of solid of composition Cs’, purer than Cs in A separates from the liquid. (Application of Lever rule.)

Now, this leftover solid is again remelted. Now, we observe that its solidification starts at temperature (T 2), higher than (T 1).

Again, the obtained solid after this process is smaller in quantity but purer in A than previous concentration.

By repeating this sequence the optimum number of times, we obtain very pure A, even though the quantity of A is very less as compared to the initial concentration of the material.

In Zone refining, this principle of phase separation is used.
The above shown figure is a model of Zone Refining mechanism. We see that the material to be purified is taken in the form of long rod. A small section of the rod is heated at a time using a coil shaped heater. The molten zone speed and width must be theoretically and practically considered depending upon the applications, the material chosen and the surrounding conditions. There are many techniques involved in heating of the zone. Induction heating is applicable to those substances that conduct electricity. An electron beam or laser beams to heat the rod are also used. The coil or beam is slowly moved from one end to another end which continuously solidifies the molten zone and melts the fresh material ahead. Hence every time we repeat the cycle, we get purer material or crystal and the ending impure part is chopped off. As seen In the previous graph, the temperature of heating coil has to be increased every time so as to obtain better results.
• In the previous slide, the picture used is from the internet.

    Please make a photo similar to that in the final document.
Zone Refining
### Step 1:

<table>
<thead>
<tr>
<th>Description of the action</th>
<th>Audio narration</th>
<th>Text to be displayed</th>
</tr>
</thead>
<tbody>
<tr>
<td>First show slides no.1 of IDD on the screen (use good effects).</td>
<td>Zone refining is a technique used to purify an element or a compound or control its composition by melting a short region (i.e., zone) and causing this liquid zone to travel slowly through a relatively long ingot, or charge, of the solid.</td>
<td>Same as text on slide 1 of IDD.</td>
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### Step 2:

<table>
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<tr>
<th>Description of the action</th>
<th>Audio narration</th>
<th>Text to be displayed</th>
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</thead>
<tbody>
<tr>
<td>Show slide no. 2 of Idd on the screen. .</td>
<td></td>
<td>FOR many semiconductor chips and other sophisticated electronic materials, elements such as germanium and silicon are needed in their ultra-pure state so as to achieve the required properties... DO you know how elements are obtained in such pure state?</td>
</tr>
</tbody>
</table>
Zone Refining
### Definitions and Keywords

- **PHASE DIAGRAM** - A phase diagram is a type of chart used to show conditions at which thermodynamically distinct phases can occur at equilibrium.
- **CRYSTALLIZATION** - Crystallization is a chemical solid–liquid separation technique, in which mass transfer of a solute from the liquid solution to a pure solid crystalline phase occurs.
- **TEMPERATURE** - *(T)*
- **LEVER RULE** - A tool which enables us to determine the relative amounts of 2 phases or the weight percentages of 2 phases in a binary phase diagram.
Zone Refining

Animation Screen

Instructions/ Working area
ZONE REFINING

Step 1

Molten Zone Containing Impurities

Purified Solid Zone

Direction of travel of heater

Impure Block

LIQUID

SOLID

LIQUID + SOLID

TEMPERATURE

T 2

T 1

a

b

c
<table>
<thead>
<tr>
<th>Description of the action/ interactivity</th>
<th>Audio Narration</th>
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<tbody>
<tr>
<td>Initially the heading “ZONE REFINING” on top appears as shown. There should be a color change continuously from 1 letter to other in the heading, i.e. Yellow color denoting flame should move from 1 letter to another and previous letter should again come back to original color, once both the words are done, cycle should again repeat. The rod drawn is actually a cylindrical rod mostly of a metal and the yellow zone shown is heater coil. So, where I have marked heater, make a burner and move it continuously along the rod with the flame as shown in the given dirn. From left to right. Initially the whole rod is of same colour but as the burner moves along, the color of previous part should change, which looks more systematic or ordered. Finally, when the frame reaches the end, show using some appropriate tool the last yellow molten part is chopped off and the flame again goes back to the start on left most size. Along with these, you can see on the right side there is circle with the no. inside, so everytime one cycle is done, change the number in that box. Also there is temp. rise in the flame every turn, so show the intensity of burner higher when the flame goes to the next turn.</td>
<td>As you can see, there is a cylindrical rod which contains element A with the impurity element B. The rod is heated with the help of a heater coil which moves from one end to the other end melting the next zone and resolidifying the previous zone just passed. Finally, the impure molten form at one end as shown is chopped off and reheating of the rod is done. The process is repeated several times till we get the material of required purity.</td>
<td></td>
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</tbody>
</table>
Now, below the rod there is a graph with 2 lines which is a binary phase diagram with the material to be obtained as A and impurity B. As you can see, I have drawn some colored lines in the graph. This runs parallel to the flame in the upper rod. When the flame starts in cycle 1, then the vertical line through ‘a’ at Cs is drawn and the line shown in white blue color slowly progresses from point ‘a’ to ‘b’ in the course of time when the flame goes from the left to right end. Now, when the last impure part is chopped off and the flame is once again ready to get started from left end, the vertical line from b to c and also downwards is drawn. Now again when the 2nd cycle starts, this graph has the red horizontal line moving from c to the other point and the cycle continues. The 2 bold lines drawn initially will remain intact. Also, don’t show varied colors for different lines, this is just for explanation purpose, so chose any color other than black and use it for all the horizontal lines show. Since, after 1 line, the 2nd horizontal line is diminished, please see to the scale of graph and draw accordingly.

The audio narration line marked in red color on previous slide should be so adjusted such that the process of chopping off and this line should coincide.

There is also a binary phase diagram shown below which runs parallel to the heating process and the graph proceeds accordingly as shown.
Explanation of Zone refining Technique
(Explanation Tab )

**Step 1:**

Show the next slide, i.e. slide no. 4.

<table>
<thead>
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<tr>
<td>Show the next slide, i.e. slide no. 4.</td>
<td>Same as on slide 5 of IDD.</td>
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</table>
Step 2:

First show the graph and then content. Show slide no. 5.

Based on a very important fact that the solid crystallizing first from the melt is purer in A or the element to be obtained than the liquid.

Same as text on slide 5 of IDD.
### Step 3:

<table>
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<th>Description of the action</th>
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</thead>
<tbody>
<tr>
<td>Show the next slide, i.e. slide no. 6.</td>
<td>Same as on slide 6 of IDD.</td>
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</table>
**Step 4:**

The above shown figure is a model of Zone Refining mechanism. We see that the material to be purified is taken in the form of long rod. A small section of the rod is heated at a time using a coil shaped heater. The coil or beam is slowly moved from one end to another end which continuously solidifies the molten zone and melts the fresh material ahead. Hence every time we repeat the cycle, we get purer material or crystal and the ending impure part is chopped off.

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<tr>
<td>first show the picture and then Show the next slide, i.e. slide no. 7.</td>
<td>The above shown figure is a model of Zone Refining mechanism. We see that the material to be purified is taken in the form of long rod. A small section of the rod is heated at a time using a coil shaped heater. The coil or beam is slowly moved from one end to another end which continuously solidifies the molten zone and melts the fresh material ahead.</td>
<td>Same as on slide 7 of IDD.</td>
</tr>
</tbody>
</table>
1.) Zone refining is based on the fact that the pure substance that has to be obtained from the impure sample has a ______ melting point than the sample.

ANS ). Lower

2.) Which of the 2 figures can not be purified by zone refining?

ANS ). FIG 1
1. Compute the percentage of solid and liquid phases for the given point B in the above phase diagram.

ANS. weight % of solid phase = 33.33 %
weight % of liquid phase = 66.67 %
Summary

• Zone refining is a technique used to purify materials, mainly semiconductors by melting a short region (i.e., zone) and causing this liquid zone to travel slowly through a relatively long rod of the solid.

• This process is based on a very important fact that the solid to crystallize first from the melt is purer in A or the element to be obtained than the liquid.

• The material to be purified by this process is taken in the form of long rod. A small section of the rod is heated at a time using a coil shaped heater. And the coil is moved from one end to another. Hence every time we repeat the cycle, we get purer material or crystal and the ending impure part is chopped off.

• Even after being a very expensive technique of purification involving low processing rates, careful handling and high energy consumption, this method is very widely used in many applications, mainly for purification of elements like silicon or germanium for electronic devices.
Links for further reading

• http://www.britannica.com/EBchecked/topic/657857/zone-melting/8129/Techniques-of-zone-refining


• Materials Science and Engineering - A first course, Prentice Hall of India Private Limited, 3rd edition by Prof. V. Raghavan.
**Step 1:**

<table>
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<tbody>
<tr>
<td>Show the text written on the slide 23 in black color, i.e. the questions first. Then make a ‘ANSWERS’ tab at the bottom of the screen. This when clicked should display the red color text, answers</td>
<td></td>
<td>First the black coloured text, and then when answers tab is clicked, the answers or the text in red colour.</td>
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### Step 2:

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<tr>
<td>Show the text in slide 24 on the screen.</td>
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<td>Text on slide 25.</td>
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### Step 3:

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<tbody>
<tr>
<td>Show the text in slide 25 on the screen similar to the way in previous slide.</td>
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<td>Text on slide 25.</td>
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**Step 4:**

<table>
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<tbody>
<tr>
<td>Show the text on slide 26. The points should be shown one by one.</td>
<td></td>
<td>Text on slide 25.</td>
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