KNSB Alloy Propellant Development

5c. Melting of KNSB Alloys:
Cure Time

Objective

The purpose of this experiment is to compare the curing rate of several Potassium nitrate/Sorbitol (KNSB) formulas that are alloyed with other sugars in varied amounts.
The % of KN is maintained at 65% The amounts of sorbitol and the alloy sugar are varied with the total sugar (fuel) content maintained at 35%. (See table in Procedures below)
This experiment is a result of the Sugar Shot to Space Propellant Development Team's Proposed Investigation 5c
Be sure to check out the Sugar Shot to Space home page

Procedure

Materials:

The Potassium nitrate (KNO_3) was fine powder (Stock #C170 - OX) from [Firefox](#)
The Sorbitol was Sorbo-Gem food grade powder
The Dextrose was anhydrous dextrose reagent grade
The Sucrose was ultrafine Baker's sugar C&H
The melting pot is a small triple batch double boiler utilizing paraffin as the heat transfer agent.

Process:

Grain size of the KNO3 was examined in the microscope. Grain size range was measured with an ocular micrometer.
Propellant batches were made in 20 grams total mass.
The ingredients for the alloy formula propellants in the table below were weighed on a triple beam balance accurate to less than 0.1gm

Designation	**Alloying sugar**	**% Ratio 1**: KNO3/Sorbitol/other	**% Ratio 2**	**% Ratio 3**	**% Ratio 4**
KNSBSU	Sucrose	65/30/5	65/25/10	65/20/15	65/15/20
KNSBDX	Dextrose	65/30/5	65/25/10	65/20/15	65/15/20

As a non-alloy control "plain" KNSB 65/35 was also prepared.
The ingredients were then mixed by passing through a fine screen. This mixture was then mixed multiple times by the "diaper method". This mixture was then placed in sealed plastic containers and mixed further by vigorous shaking. Each of the 20 gram batch formulas was then melted in a double boiler set up that uses melted paraffin as the heat transfer medium. The double boiler set up prevents any chance of an ignition hot spot in the melting process. The oil bath was set to a temperature over the melting temperature of the higher melting sugar.

- **Figure 1 For the previous experiment PDT - 5a the propellant was mixed with the thermometer as the stir stick and temperatures were**

recorded as the propellant melted. Consistency of the slurry was recorded. (see PDT-5a.)

- **Figure 2 The propellant was poured into 24mm "Bates" grains for the purpose of investigating cure rate for this experiment 5c. The grains were capped with washers to prevent accumulation of moisture on the surface. See grains in figure 3:**

- **Figure 3**

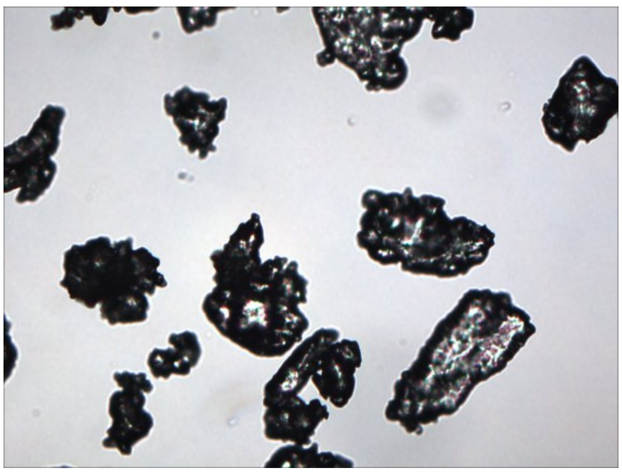
Method of measuring cure rate:
The curing process occurs as a gradation of firmness so to get some kind of standardization I would push a sewing pin hard enough to be a bit painful to the pushing finger for one full second. If the pin could no longer penetrate the grain at least 1mm in one second then I called that point in time "cured" The cure timing started when the grain reached about 37C (body temp) and timing stopped when the grain reached the "Pin prick" criteria mentioned above. Timing was measured to the nearest minute until 15 minutes had passed. Then the grain was checked every 5 minutes until 30 minutes had passed. If the grain was still not cured at 30 minutes then it was checked every 30 minutes until 8 hours had passed. At the 8 hour point only the non-alloy control had not cured (and I went to bed :-) In the morning at 15 hours it had cured. At that time I could insert the pin only about 0.5mm so 15 hours was recorded as the control cure time. Prior to starting the curing time measurements the grains were timed to see how long they would take to cool. For the reasoning on seperating these time periods see the discussion section

Results

Crystal size analysis:

KNO3 crystals:

The majority of the KNO3 crystals measured 20 microns to 200 microns
The smaller particles tended to stick to the larger ones



Alloy Cure Rate Data:

ALLOY	% FORMULA	TIME TO COOL	TIME TO CURE *
KN/SB	65/35	15 minutes	>8 hours & <15 hours
KN/SB/DX	65/30/5	15 minutes	3.5 hours
KN/SB/DX	65/25/10	15 minutes	2.0 hours
KN/SB/DX	65/20/15	15 minutes	0.5 hours
KN/SB/DX	65/15/20	15 minutes	10 minutes
KN/SB/SU	65/30/5	10 minutes	20 minutes
KN/SB/SU	65/25/10	10 minutes	15 minutes
KN/SB/SU	65/20/15	10 minutes	8 minutes
KN/SB/SU	65/15/20	10 minutes	6 minutes

* This time was measured starting when the grain reached ~37C and stopped when the grain reached "pin prick" criteria.

Discussion

As mentioned earlier, the total curing time was seperated into two time measurements. See Table above. The cooling time and then the curing time (post cooling)
The reason for this is because the 24mm grains in this experiment are very small and cool much faster than larger grains. During our local sorbitol grain pouring meetings I have sometimes made 24mm sorbitol grains with the excess melt after we have poured larger 54mm grains for the VAROOM hpr-EX group. The 24mm grains (made later) will cool to room temp while the large grains (made earlier) will still be warm to hot.
This cooling time difference would only be greater with even larger grains.

Heat is lost at the surface of the grain to the air and surroundings. A larger grain will have less surface area per unit of mass than a smaller grain. For example a mass filling 1 cubic centimeter (cc) of volume will have 6 square centimeters (6 cm2) surface area per one cubic centimeter of mass/volume. (Assume density is the same so I can use volume in place of mass)
So that is 6 cm2/cc for a 1 cm cube.
Now consider a 10 cm high cube. That is 600 cm2/1000 cc or 0.6 cm2/cc
That is 10 times less surface area per unit mass.

Also one must consider when the center of the grain will be cool, not just the surface. The heat in the center portion of a large grain must conduct further just to get to the surface.
The grains in this test are only 17mm wide and have a core of 5mm. This gives them a web-thickness (TWEB) of only 6mm.

Once cooled the cure rate time values should correlate to grains of various sizes.
However it was noted that the small 65/35 plain sorbitol grain cured in about 15 hours. Yet at Richard Nakka's site and an email discussion with Bill Colburn it is clear that larger sorbitol grains take ~24 hours just to possibly be firm enough to remove the mandrel and another day to cure completely.
I think that due to my small 6mm TWEB that these 24mm grains represent the curing seen on the "surface" of larger grains.
Despite all these caveats for the variable of size (factor of scale) this 24mm grain alloy data is of value. Within this experiment the variable of scale is removed. One can compare one alloy to another and to the control 65/35

KN/SB. Then knowing how KN/SB cures for their size grains one can extrapolate how the alloy would probably cure at that size.

See all the alloys in comparison to the control in Graph 1:

- **Graph 1**

For more detail on the alloys see Graph 2:

- **Graph 2**

Just the anhydrous dextrose alloys are seen in Graph 3:

- **Graph 3**

Just the sucrose alloys are seen in Graph 4:

- **Graph 4**

Conclusions

The anhydrous dextrose alloys appear to provide the best range for controlling cure rate. It should be fairly easy to tailor the correct % of dextrose to provide the desired cure rate. However if it is desired that the propellant fuel be predominantly sorbitol then sucrose can provide cure rate control with very low percentages of sucrose. If sucrose does seem the best alloy sugar choice then it would be interesting and helpful to try this same experiment with 1%, 2%, 3%, and 4% sucrose alloys. Cure rates closer to an hour or two might be possible with these low amounts of sucrose.
If this is attempted then I would think that very fine sucrose very well mixed would be important to provide homogeneity for the curing of the grain. The Baker's sugar I used is finer than table sugar and avoids the starchy additives sometimes found in powdered sugars.
One advantage to sucrose in low percentages is that it should be easier to avoid any degradation/caramelization. In experiment PDT-5a I had trouble with yellow degradation in the 20% dextrose formula. See figure 3 below.
However the cold weather and small scale set-up I used that day contributed to a lack of control of maximum oil temp. 20% dextrose should be do-able in a larger volume with tighter temperature control?

Depending on the alloy's effect on motor performance these alloys could provide much benefit when pouring grains on-site. Rocketeers could possibly avoid the 48 hour cure time and still have the basic performance of sorbitol? To prove this would require making some larger grains and characterizing the formula further.

(That is beyond what my dinky little Drysophila sized set up can do :-)

Figures and Graphs

Figure 1:

Three formula set up

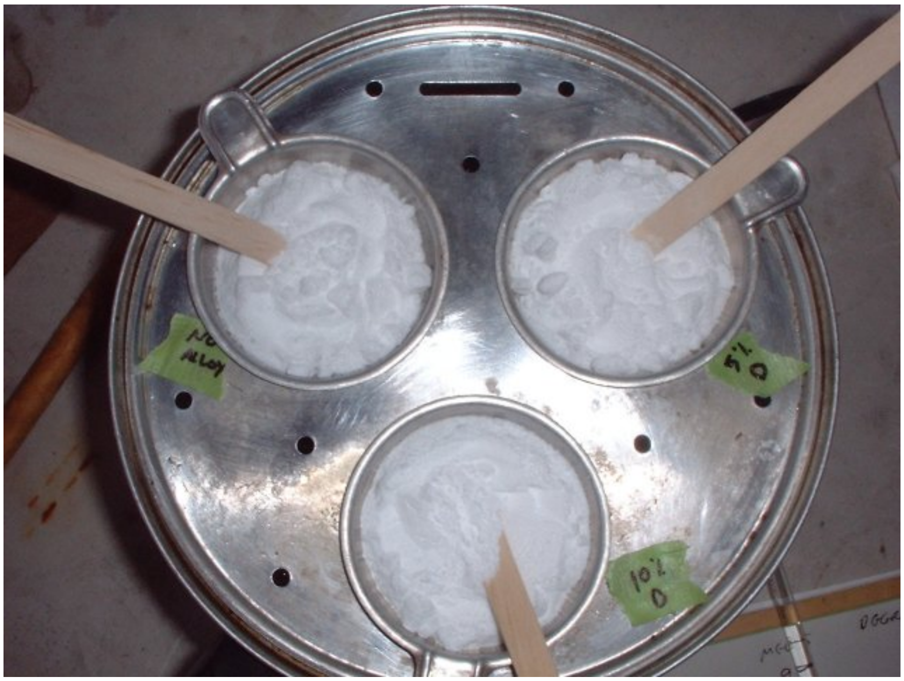
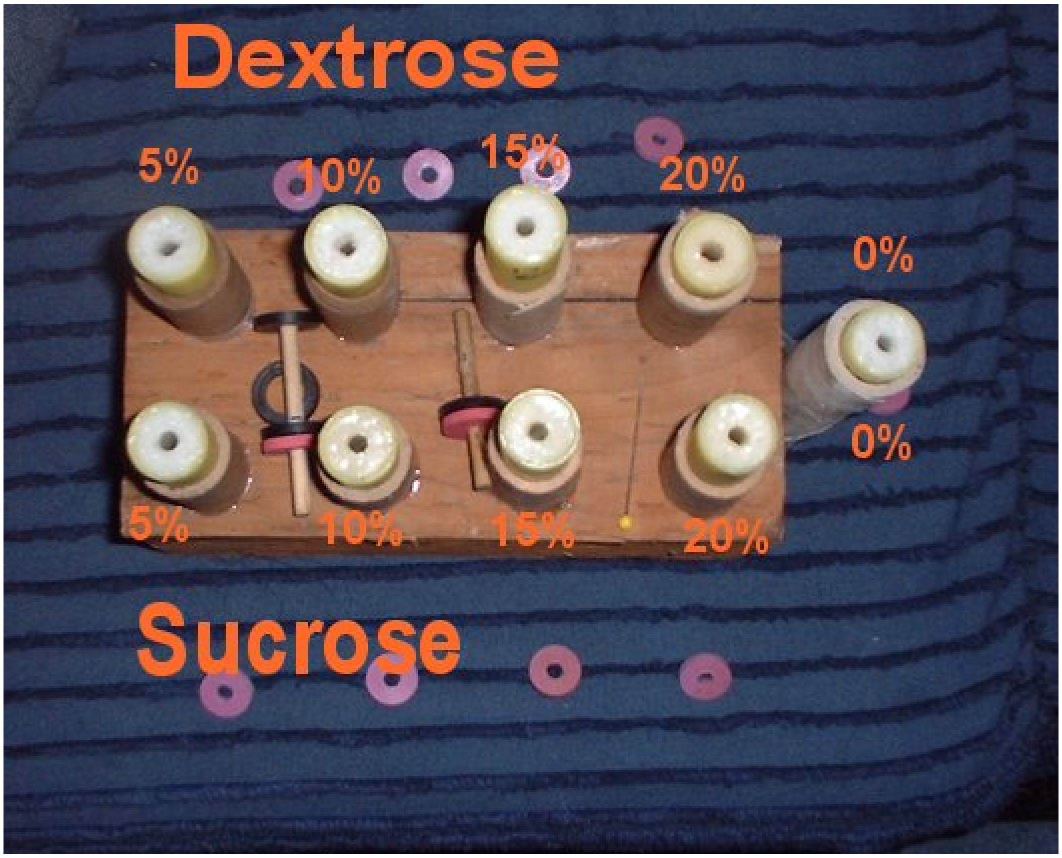


Figure 2:

Temperature recorded during melt;

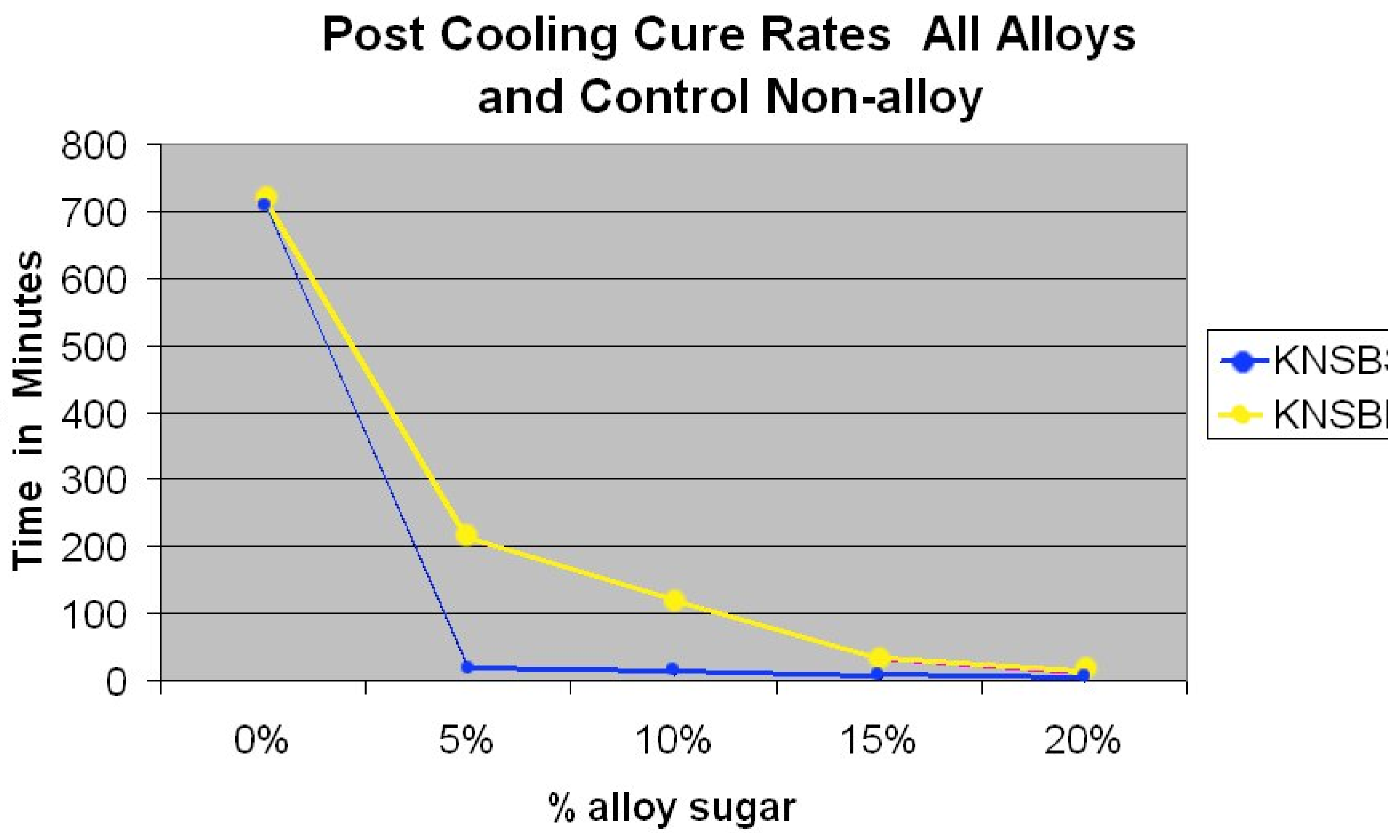
Figure 3:

24mm Grains; Note yellowing on 20% dextrose formula.



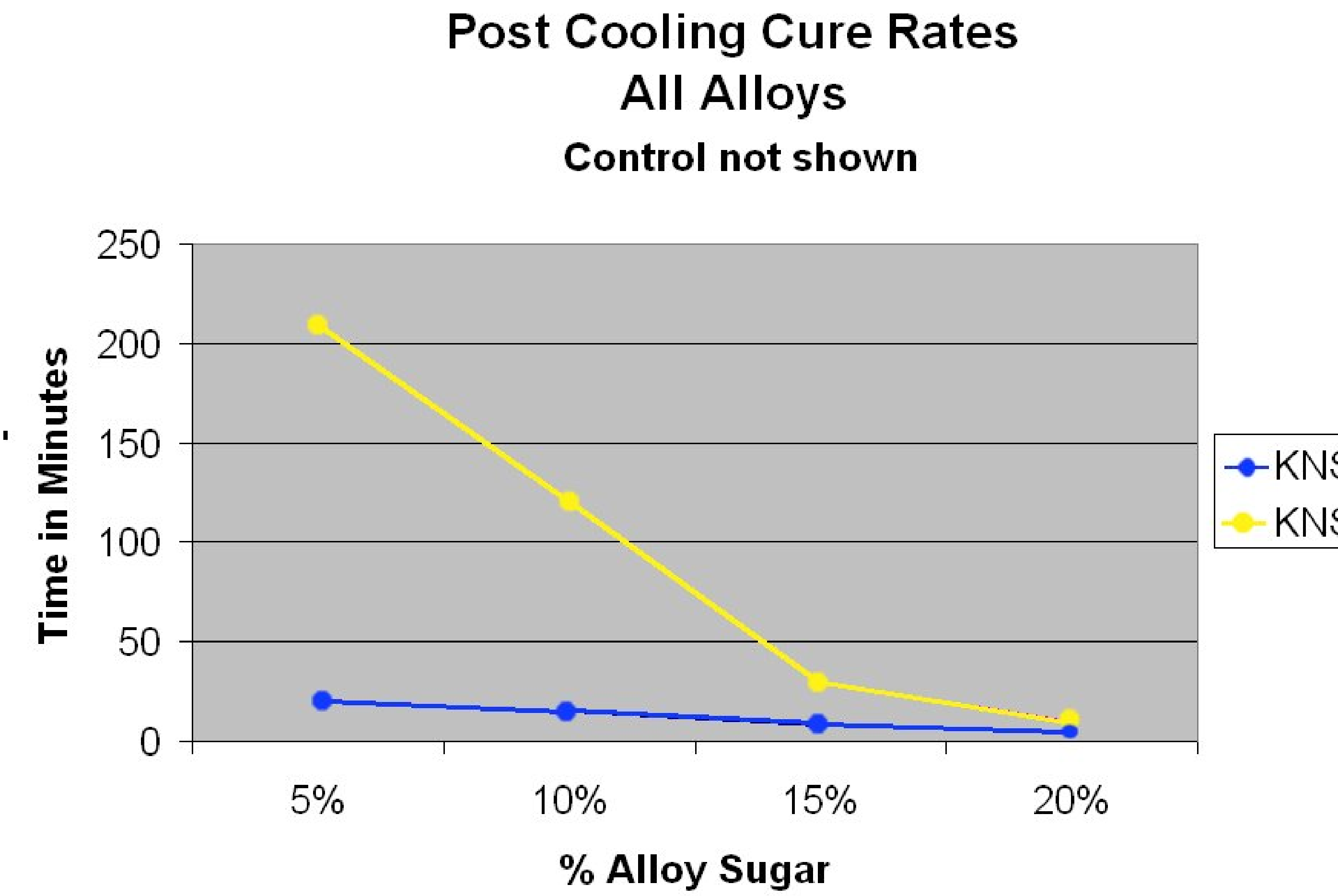
Graph 1:

KNSBDX vs KNSBSU Alloys with 0% alloy control also graphed;



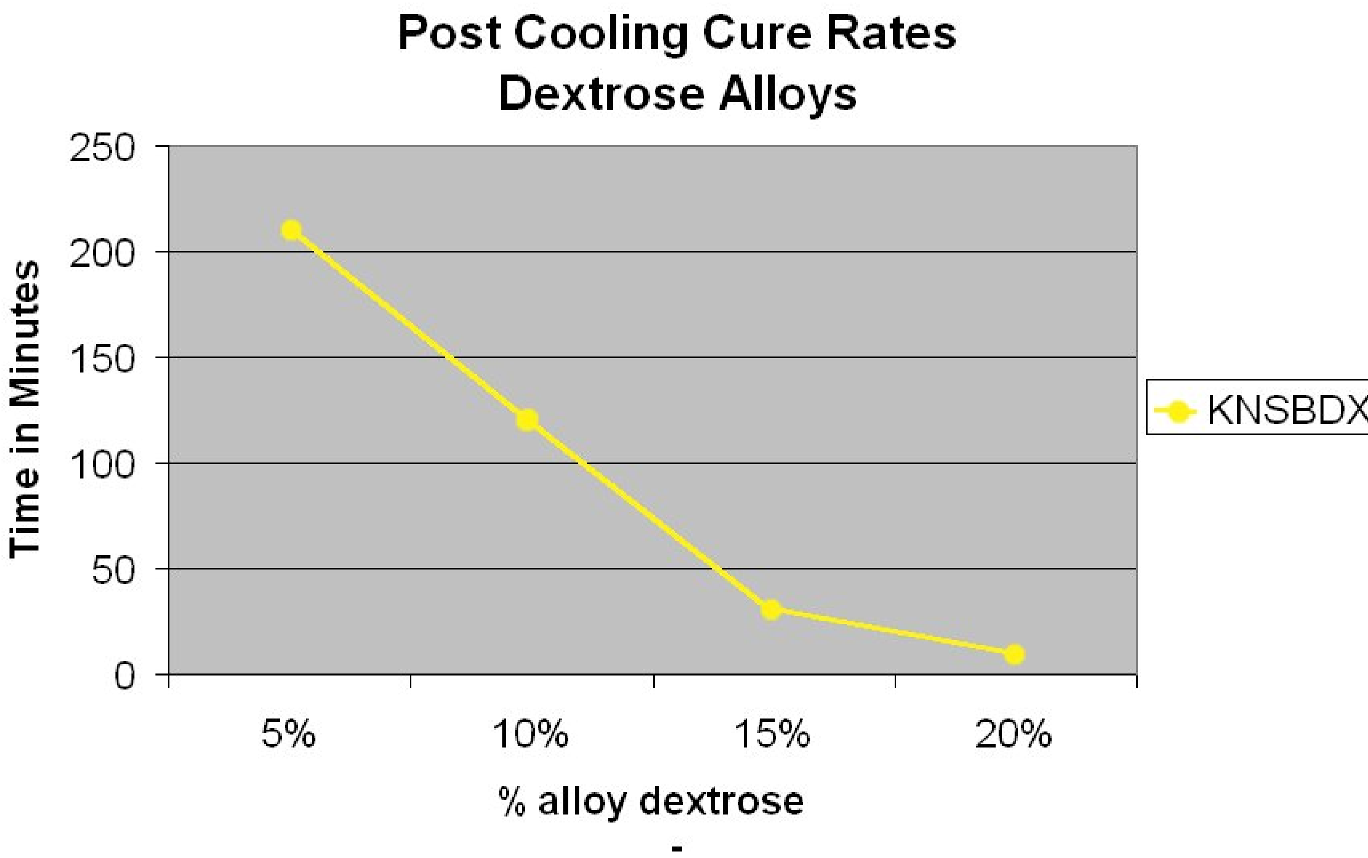
<u>**Graph 2:**</u>

KNSBDX vs KNSBSU Alloys; Control not graphed;



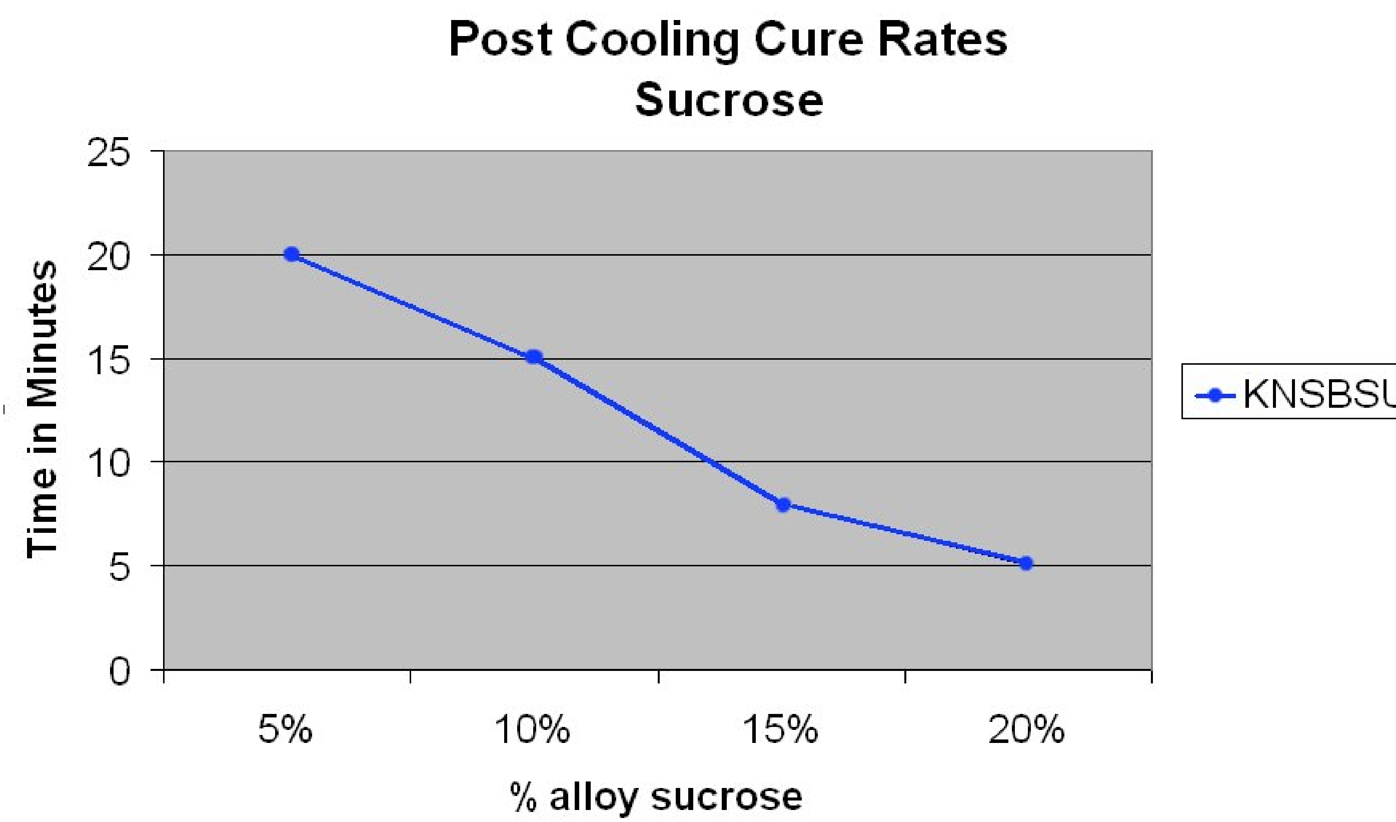
Graph 3:

KNSBDX Alloys; Control not graphed



Graph 4:

KNSBSU Alloys; Control not graphed



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