NON-DESTRUCTIVE TESTING OF BORON NITRIDE

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ABSTRACT

Hexagonal boron nitride (BN) is the material of choice for manufacturing break rings for the horizontal continuous casting of steel. Due to the criticality of this application, only a 100% acceptance level can be tolerated. A non-destructive testing method was successfully developed to accomplish this. Comparison of this method with more traditional non-destructive tests showed it to be comparable.

INTRODUCTION

The idea of horizontally continuous casting has been around since Henry Bessemer patented his concept in 1857. Nevertheless it has only been recently that it has become technically feasible to do so. The first commercial horizontal continuous caster for steel was developed by General Motors in the 1960s.[1] After the shut down of this installation, two decades had to pass before another machine was brought on line for the casting of steel. The original equipment and all subsequent ones have a critical component called a break ring which is the last refractory the molten steel sees before it starts to solidify. The break ring's function is to present a consistent position for the solidifying steel front. In this role, it allows for the mechanical separation of the solidified steel shell from the refractory. In order to accomplish this, the break ring has to be non-wetted by the steel and avoid all chemical interaction with the metal. In addition, this part has to meet some stringent physical properties. In order of importance, they are:

a) excellent thermal shock resistance
b) high thermal conductivity
c) volume stability at high temperature
d) ease of machinability

Hexagonal boron nitride (BN) meets all of the above criteria and therefore is the material most commonly used in this application.[2,3,4]

The manufacture of boron nitride is complex and time-consuming and can easily give rise to product inconsistencies. Because of this, break ring failures during casting have occurred. As a consequence, the need arose to develop a means of eliminating failures. As a failure means an automatic halt to the casting process with the concomitant expense of time and materials, it is something that has to be avoided. This paper will describe a non-destructive testing method for boron nitride break rings and compare it to more traditional tests.

BACKGROUND

In early 1984, a horizontal continuous casting machine for the casting of stainless and alloy steels was started up at Baltimore Specialty Steels Corporation, a wholly owned Armco subsidiary. This machine is capable of casting billets of varying sizes and each size requires a break ring with its own characteristic dimensions. A few years after startup, a rash of break ring failures occurred during casting. As this was an unacceptable situation, the decision was made to attempt to discriminate between "good" and "bad" rings in a non-destructive way. Some work had already been done in that area[5] and it was assumed that the test method, although previously only used on an extremely limited sample base, would indeed be applicable to this problem.

MANUFACTURE

At least nine proven ways of manufacturing boron nitride powder exist. The reaction basically consists of nitriding a boron containing compound (CaB_6, B_2O_3, H_3BO_3, etc.) at high temperature according to the following formula:

$$2B + N_2 \xrightarrow{\geq 900\,°C} 2BN$$

Different raw materials yield varying amounts of boron nitride mixed with impurities. The resulting mixture may require extensive grinding and re-nitriding before going to completion.

The reaction

$$B_2O_3 + 2NH_3 \rightarrow 2BN + 3H_2O$$

provides a cheap and convenient production method. The end product is commercial boron nitride powder and although containing no alcohol soluble material, i.e. no free B_2O_3, at room temperature, produces B_2O_3 when heated above 1200°C in an inert atmosphere.[6] This characteristic allows it to be easily hot pressed at temperatures as low as 900°C.[7,8,9] It should be noted that B_2O_3 is necessary for hot pressing as it acts as the binder and imparts cold strength to the body. The problem is that the amount of B_2O_3 present in the final product can be highly variable leading to erratic wear. This can lead to the premature failure of the break ring.

In general terms, hot pressing consists of filling a graphite mold with boron nitride powder which is then subjected to pressure and temperature for a fixed length of time. The resulting solid is called an ingot or slug. A fixed number of break rings can be cut out of each ingot, the limiting factors being their thickness, diameter and geometric configuration.

EXPERIMENTAL PROCEDURE AND RESULTS

Two different sets of non-destructive tests were run: On complete break rings 155mm and 100mm square in size, as well as on boron nitride bars cut out of these break rings. All boron nitride break rings used in this study came from the same supplier.

As the main purpose of this work was to develop a testing procedure for complete break rings, considerable work was put into this phase. Preliminary efforts were reported in 1985.[5] At that time, two different non-destructive tests were performed on whole 100mm square break rings. Initial work consisted of ultrasonic velocity measurements.*1 This technique measures the time it takes a sound wave to travel through the sample. A 19mm diameter probe size was used. The input signal's frequency was 50 Hz. A coupling gel was applied to the transducer and the receiver for optimizing contact. The number so obtained can be used to calculate the modulus of elasticity. Figure 1 shows all the points where measurements were taken. The measurements through the thickness of the sample were soon discontinued as no variability could be detected, even in specimens with known flaws. The longitudinal measurements did show some variability, but most of it could be attributed to length difference among the samples. It was later discovered that the ultrasonic velocity method cannot be used for a donut-shaped body as two opposite traveling wave fronts are setup in the body which interfere with each other.

Another non-destructive test tried was based on shock excitation.*2 This method makes use of a piezoelectric transducer which picks up a sample's fundamental vibration brought about by a light tap on it. Although this technique was sensitive enough to show differences among the break rings, the number of samples available, 28, precluded the determination of their statistical significance. Figure 2 is a histogram of the data obtained. Note that this is raw data, not yet converted to modulus of elasticity. It includes the values for rings with known flaws. These questionable rings usually gave raw readings greater than 940.

Based on these results, the above test procedure was taken into the field to test whole rings before they were put into service. The purpose was twofold: to increase the data base and to determine if the instrument could also discriminate between "good" and "bad" 155mm break rings as the caster was experiencing problems with that size. As a consequence of the unusual size of the break rings which were causing the problem, only two definitely unacceptable rings existed. Test data from these two rings were used as the upper limiting value for acceptable. The raw data obtained showed that this method was sufficiently sensitive to discriminate between acceptable and unacceptable rings (Figure 3). In addition, sorting of the values so obtained by ingot number, i.e. production lot, showed that they could be either all good, all bad or mixed (Table I). The lots with mixed numbers were declared unacceptable due to the inconsistency in their properties as reflected by the non-destructive test data. Checking the experimental values against actual caster production logs on break ring performance confirmed the conclusions arrived at through testing. Figures 4 and 5 show some of the actual field data generated for 100mm and 155mm rings.

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*1 James V-meter
*2 Grindo-Sonic

Although good correlation between field data and raw shock excitation values existed, no attempt had been made to correlate those values with the more traditional non-destructive test methods. In order to remedy this gap, boron nitride break rings were cut into bars and their modulus of elasticity was determined using the ultrasonic velocity method, the shock excitation method, and the resonant frequency method (ANSI/ASTM C747). Although this last method was written for carbon and graphite, that presented no problem as boron nitride's crystallography is almost identical with graphite's[10] to the point that it is called "white graphite."

The resonant frequency method consists of inducing a vibration in the specimen with a phonograph record needle and detecting those frequencies with another phonograph needle. The specimen has to be supported so that it is free to oscillate. A search is made for its fundamental frequency of vibration which can be read off any suitable meter. Using appropriate formulae, this number is then converted into the modulus of elasticity.

Two bars from each ring were subjected to all three tests and results are shown graphically in Figure 6. The values obtained for each pair are plotted next to each other so that two consecutive numbers represent one ring. As bars from both 100mm and 155mm break rings were used, the four values obtained for the longer bars were plotted at the end of the graph. Table II lists all the modulus of elasticity values obtained. From all the data, it can be seen that the three non-destructive methods closely parallel each other with the values obtained by ultrasonic velocity measurements being consistently lower than those obtained by the shock excitation or the resonant frequency method.

In order to determine if the non-destructive test methods could be correlated to strength, as is generally assumed, cold modulus of rupture tests were run according to ASTM C133. The results from the cold modulus of rupture test are also shown in Table II.

DISCUSSION

It is an established fact that the ultrasonic velocity and the resonant frequency methods are comparable and can be used for refractories[11,12] and graphites.(13) No such work had been carried out on fine grained ceramics and only very limited work had been done using the shock excitation method.[14] This work has shown the eminent feasibility of adding this method to the list of non-destructive tests. A further advantage is that the geometry of the break ring is of no consequence allowing for non-destructive testing in the field.

Due to the good correlation of the whole ring shock excitation values with the actual caster records, upper limits have been worked into the product purchase specification. These have had to be rewritten several times to reflect all the data which has been accumulated over the years. Even so, no information has been gleaned on a lower bound for these values. In addition, close cooperation between Armco and the boron nitride manufacturer has allowed them to develop an internal product specification whose results meet our needs. This has assured a 100% defect-free supply of break rings.

ACKNOWLEDGEMENTS

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Table I

Shock Excitation Values for 150mm Square
Break Rings Arranged by Lots

"Good"	"Bad"	"Mixed"
1599	1780	1667
1623	1795	1704
1606	1809	1776
1595	1812	1769
1602	1796	1793
1592	1787	1799
	1785	1810
		1635
		1676

Table II

Cold Modulus of Rupture Values and Modulus of Elasticity as Obtained Using Three Different Non-Destructive Tests

Sample I.D.	MOE by Shock Excitation (kPa x10^6)	MOE by Ultrasonic Velocity (kPa x10^6)	MOE by Resonant Frequency (kPa x10^6)	Cold Modulus Rupture (kPa)
A-1a	61.90	53.82	62.04	33701
A-1b	62.35	55.88	61.67	
A-2a	66.45	58.56	66.93	29695
A-2b	62.33	55.50	61.07	
A-3a	64.93	58.88	63.56	
A-3b	65.49	59.49	63.75	
A-4a	52.02	44.31	51.13	
A-4b	50.97	45.15	49.83	
C-1a	55.45	51.67	55.76	28649
C-1b	56.36	54.75	56.63	
C-2a	47.80	40.27	46.67	29903
C-2b	47.48	39.83	47.06	
C-3a	50.32	44.05	49.78	
C-3b	47.91	42.30	47.73	
C-4a	49.93	42.48	49.72	
C-4b	49.11	41.05	48.45	
D-1a	64.94	58.49	66.09	43972
D-1b	67.82	60.90	69.71	
E-1a	65.71	58.02	65.68	44127
E-1b	66.11	58.09	66.19	

Note: Samples A and C were 100mm long.
Samples D and E were 155mm long.

FIGURE CAPTIONS

FIGURE 1 - Schematic of a boron nitride break ring showing all the points where ultrasonic velocity measurements were taken.

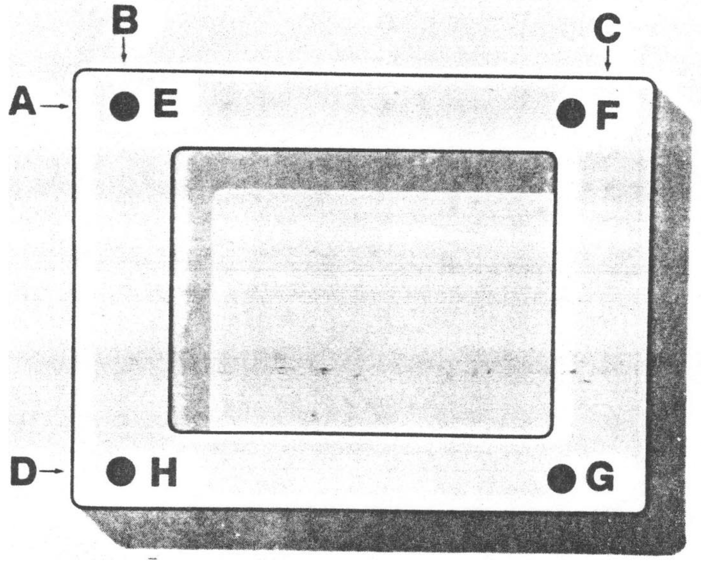
FIGURE 2 - Histogram of experimental shock excitation values for 100 mm square break rings.

FIGURE 3 - Histogram of experimental shock excitation values for 155 mm square break rings.

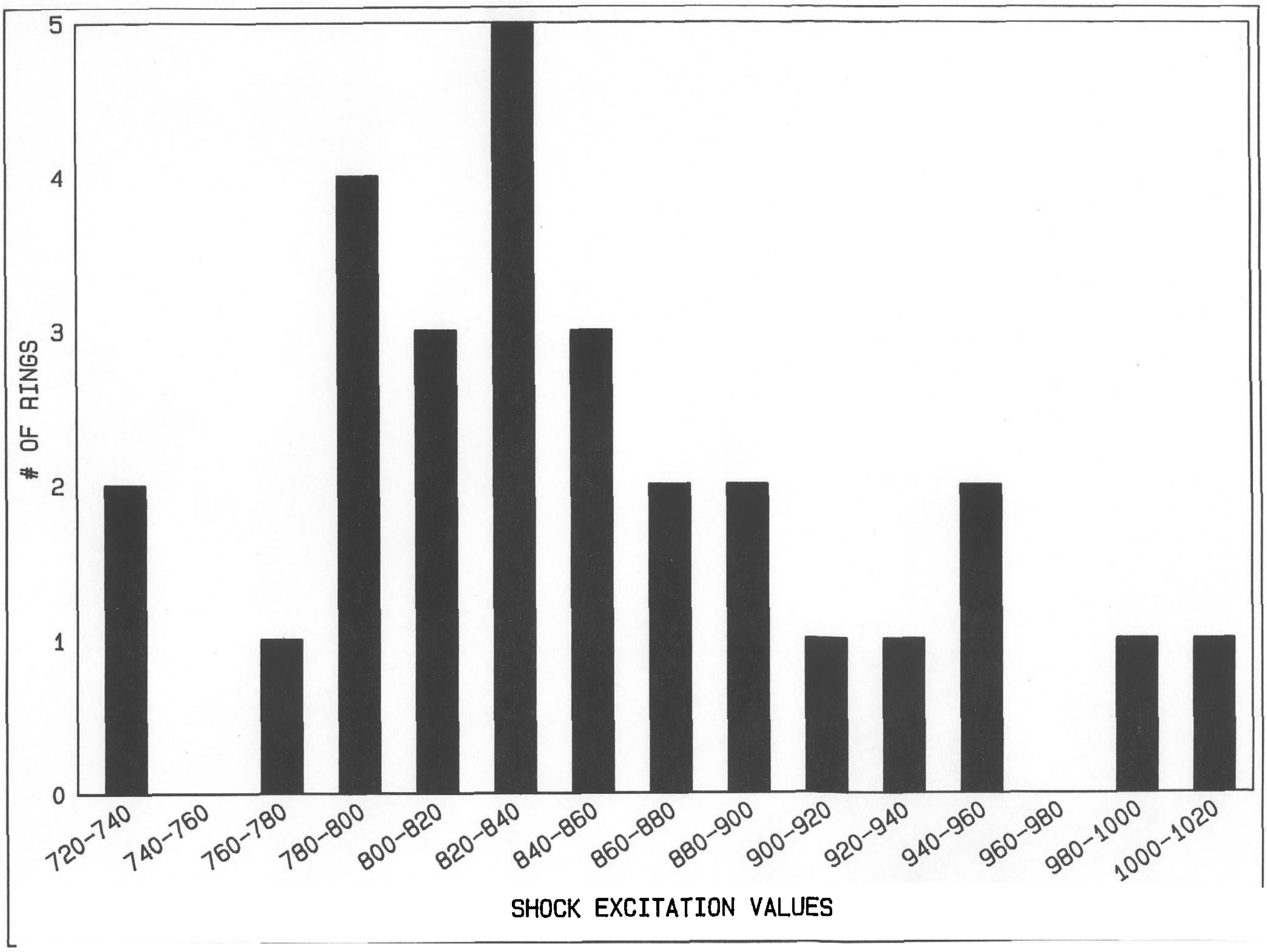
FIGURE 4 - Field shock excitation values plotted as a histogram (100 mm square break rings).

FIGURE 5 - Field shock excitation values plotted as a histogram (155 mm square break rings).

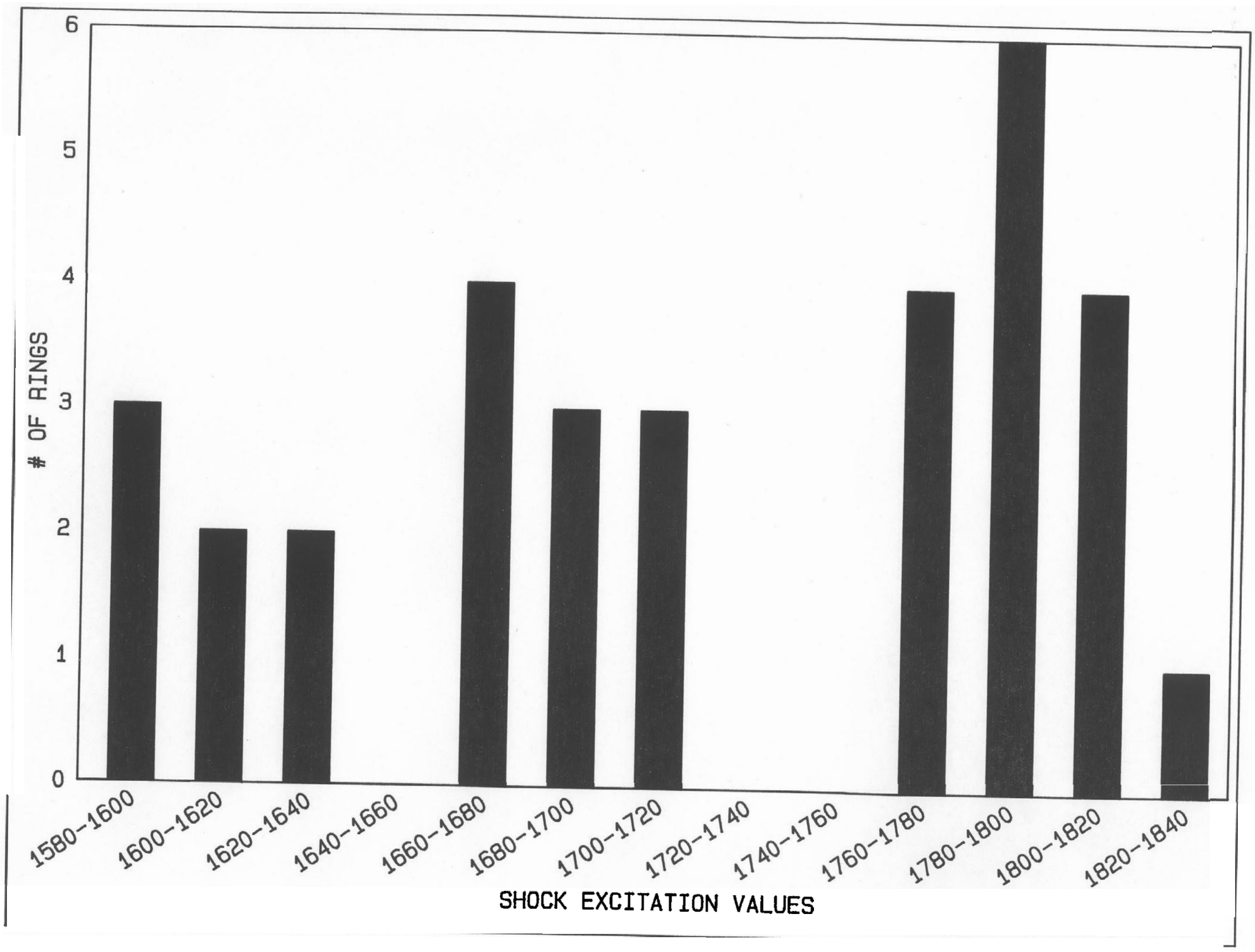
FIGURE 6 - Comparison of the modulus of elasticity using three different methods on the same samples.



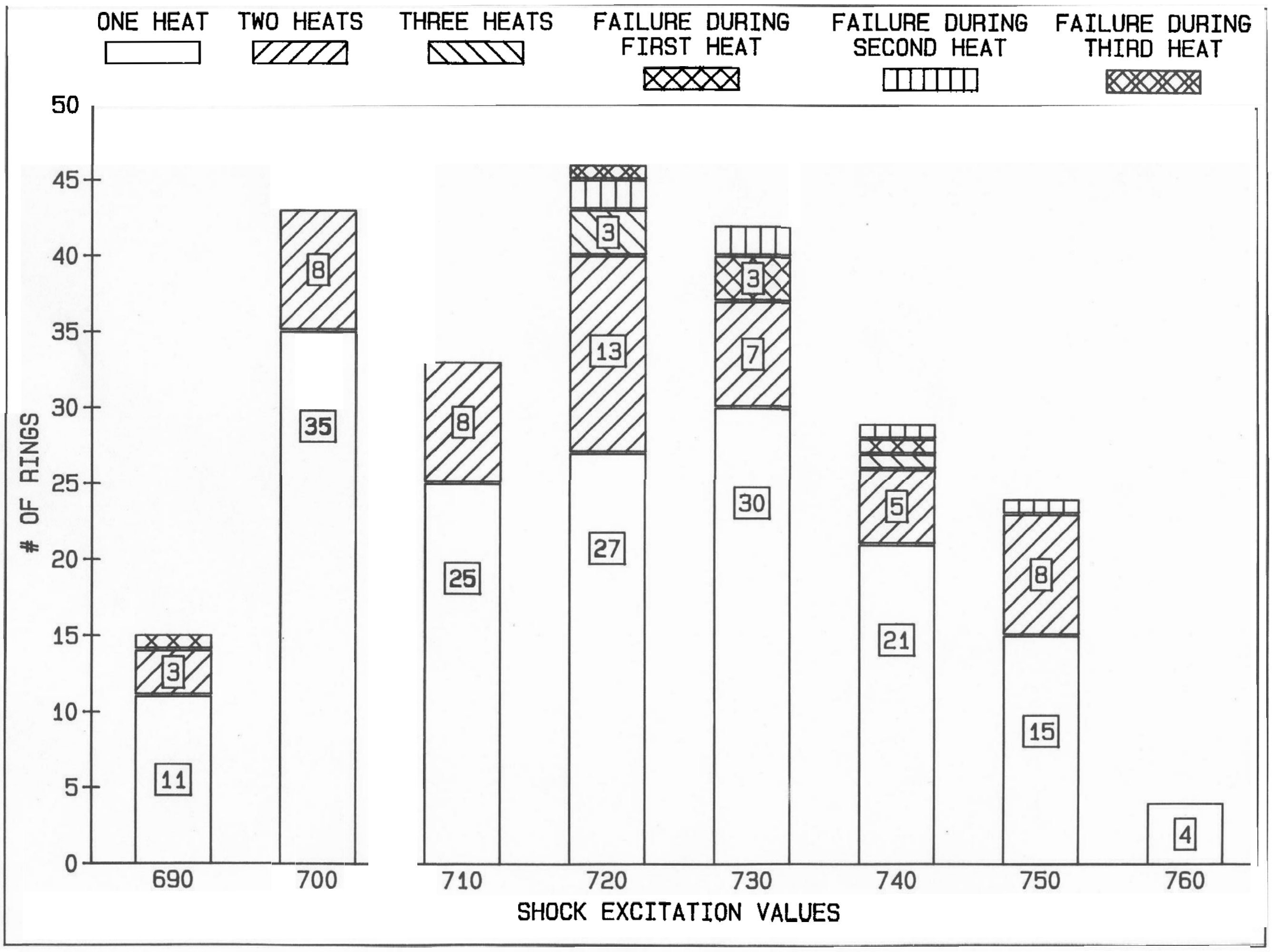
B
C
A
E
F
D
H
G



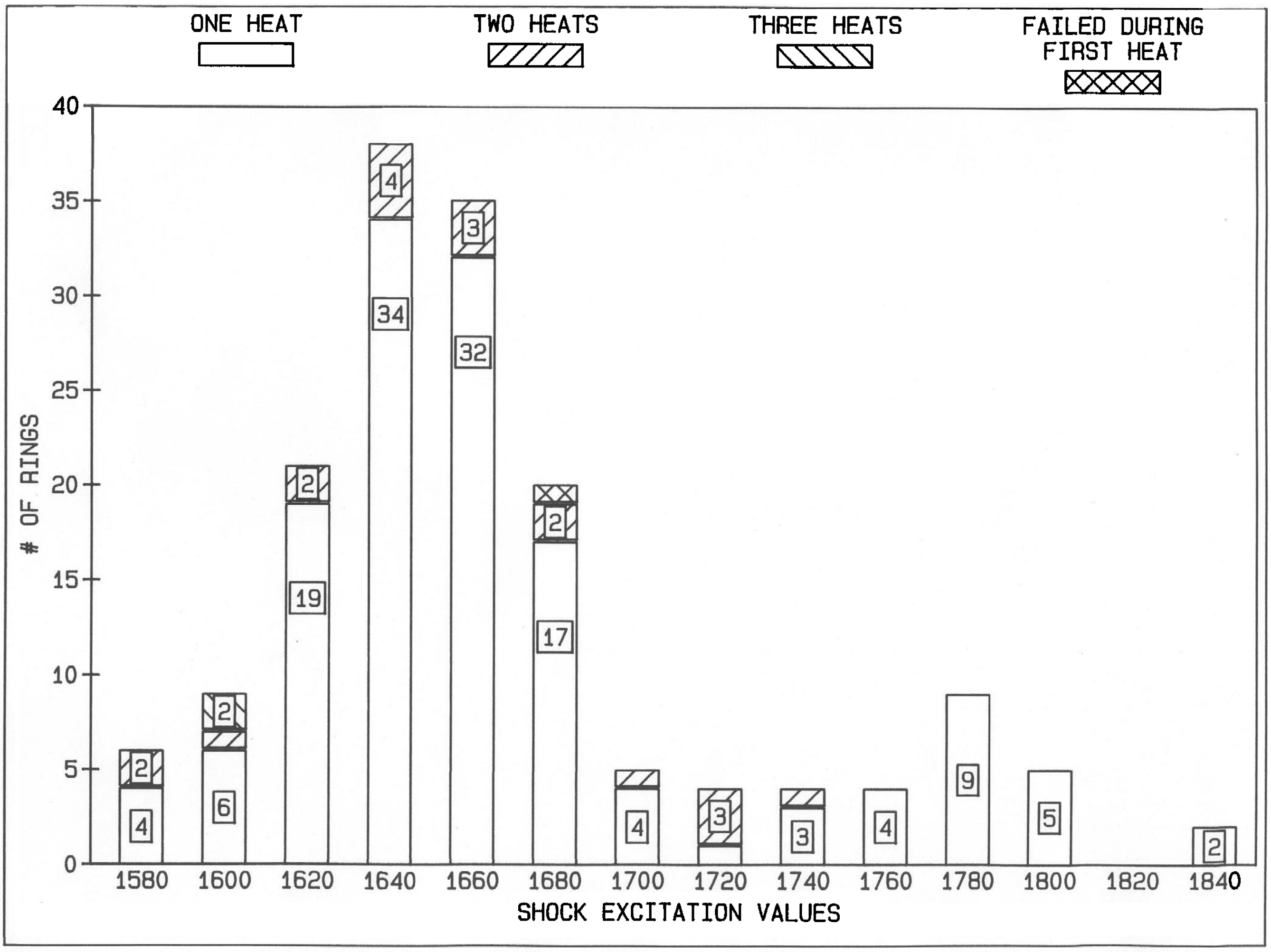
OF RINGS
0
1
2
3
4
5
720-740
740-760
760-780
780-800
800-820
820-840
840-860
860-880
880-900
900-920
920-940
940-960
960-980
980-1000
1000-1020
SHOCK EXCITATION VALUES

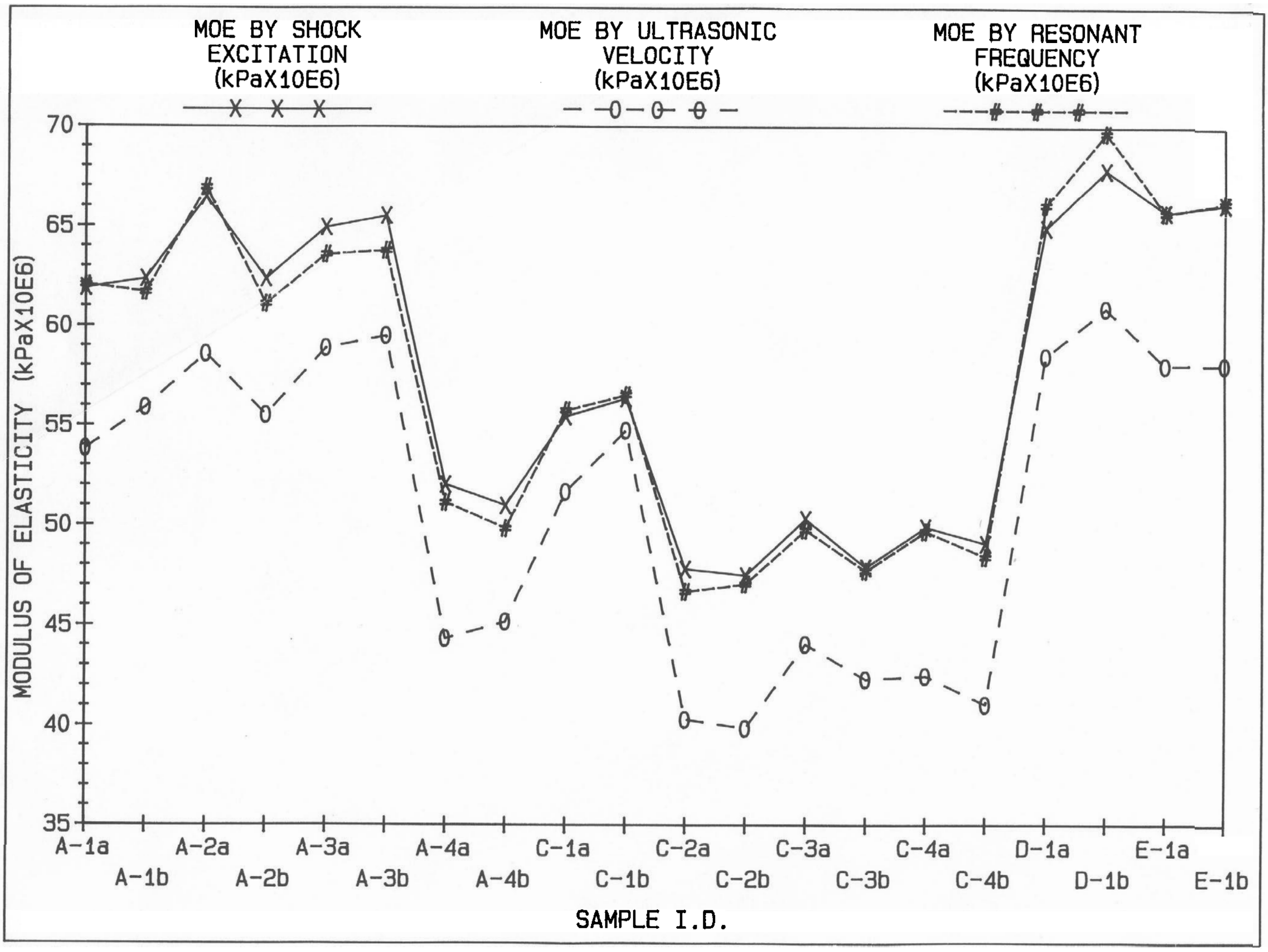


OF RINGS
0
1
2
3
4
5
6
1580-1600
1600-1620
1620-1640
1640-1660
1660-1680
1680-1700
1700-1720
1720-1740
1740-1760
1760-1780
1780-1800
1800-1820
1820-1840
SHOCK EXCITATION VALUES



ONE HEAT
TWO HEATS
THREE HEATS
FAILURE DURING FIRST HEAT
FAILURE DURING SECOND HEAT
FAILURE DURING THIRD HEAT
OF RINGS
SHOCK EXCITATION VALUES
0
5
10
15
20
25
30
35
40
45
50
690
700
710
720
730
740
750
760
11
3
35
8
25
8
27
13
3
30
7
3
21
5
15
8
4


ONE HEAT
TWO HEATS
THREE HEATS
FAILED DURING FIRST HEAT
OF RINGS
40
35
30
25
20
15
10
5
0
4
34
3
32
2
19
2
17
2
6
2
4
4
3
3
4
9
5
2
1580
1600
1620
1640
1660
1680
1700
1720
1740
1760
1780
1800
1820
1840
SHOCK EXCITATION VALUES


MOE BY SHOCK EXCITATION (kPaX10E6)
MOE BY ULTRASONIC VELOCITY (kPaX10E6)
MOE BY RESONANT FREQUENCY (kPaX10E6)
MODULUS OF ELASTICITY (kPaX10E6)
70
65
60
55
50
45
40
35
A-1a
A-1b
A-2a
A-2b
A-3a
A-3b
A-4a
A-4b
C-1a
C-1b
C-2a
C-2b
C-3a
C-3b
C-4a
C-4b
D-1a
D-1b
E-1a
E-1b
SAMPLE I.D.