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"Análisis del Desgaste en Brocas Esféricas de Diamante de una Fresa Odontológica"

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Resumen

Se presenta un trabajo de investigación para analizar cuantitativamente el desgaste de brocas esféricas de diamante. Para simular lo más fielmente posiblemente las condiciones de uso clínico, se diseñó un experimento de laboratorio para comparar brocas de dos marcas diferentes, empleadas en intervalos de 30min para preparar cavidades en piezas dentales extraídas. Se cuantificó el cambio de masa de cada broca y el cambio en su contorno externo. Además, mediante análisis microscópico, se observaron y detallaron los distintos mecanismos de desgaste que intervienen durante el uso de las brocas. Finalmente, se comparó la tasa de desgaste de cada broca y su costo unitario.

Abstract

This research work is presented as a quantitative analysis of wear generated on spherical diamond dental burs. To reproduce the clinical conditions in the most precise manner, a laboratory experiment was designed, in which burs from two different manufacturers were compared, each of them being used during 30-minute intervals in cavity preparations on extracted dental pieces. The change in mass and in the outer profile of each bur was quantified. In addition, by means of microscopic analysis, the different wear mechanisms involved during the use of the burs were observed and described. Finally, a comparison was made between the burs' wear rate and unit price.

Acta de Entrega y Constancia de Defensa Pública

Se certifica que se ha recibido el Informe Final de la Práctica Dirigida, realizada por el estudiante Juan José Alvarado Hernández, carné 201016689, la cual se titula: “Análisis del Desgaste de Brocas Esféricas de Diamante de una Fresa Odontológica”, y que se realizó en el II Semestre de 2016.

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Mención especial merecen todos los que de una u otra forma aportaron a este trabajo con la ciencia del vivir y la ingeniería emocional. Un profesional no se forma únicamente con conceptos técnicos sino con calidez humana. Gracias por mantenerme con los pies en la tierra.

Dedicatoria

A don Jorge Arturo Mata, Erick Sánchez y Marvin Rodríguez, cuyo trabajo es la fiel definición de un servicio abnegado, humilde y profesional; sus nombres deberían engalanar la primera página de copiosos documentos.

Cualquiera, pues, que me oye estas palabras, y las hace, le compararé a un hombre prudente, que edificó su casa sobre la roca. Descendió lluvia, y vinieron ríos, y soplaron vientos, y golpearon contra aquella casa; y no cayó, porque estaba fundada sobre la roca. Pero cualquiera que me oye estas palabras y no las hace, le compararé a un hombre insensato, que edificó su casa sobre la arena; y descendió lluvia, y vinieron ríos, y soplaron vientos, y dieron con ímpetu contra aquella casa; y cayó, y fue grande su ruina.

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1. Introducción

Los instrumentos utilizados en procedimientos odontológicos se exponen a diversos ambientes y procesos, que remiten directamente en su funcionalidad y vida útil. Muchas variables son las que afectan el funcionamiento de estos equipos, tanto técnico/operativas como estomatognáticas (del griego *στόμα*, boca, y *γνάθος*, maxilares). Es por esto, que las técnicas modernas en salud bucodental emplean diversos aparatos que varían en material, tamaño, geometría y función.

De entre los más diversos equipos, cabe destacar que los más frecuentemente empleados son aquellos destinados a desgastar, tallar, cortar, desbastar y pulir piezas dentales. Algunas técnicas modernas, como el corte por láser, han intentado sustituir los equipos mecánicos rotatorios clásicos, aunque su alto costo de fabricación no ha sido bien recibido por los especialistas y el elevado consumo de energía, podría poner en riesgo la seguridad de éstos y sus pacientes, al generar temperaturas tan elevadas. De darse la sustitución de estas técnicas, podría tomar décadas. Así, conviene dedicarse a comprender más profundamente el comportamiento de los aparatos más tradicionales, en especial su interacción con el entorno biológico en el que operan, ya que esto podría proporcionar mejoras en los procedimientos y en los resultados obtenidos actualmente, sin la necesidad de invertir en nuevos equipos que pueden poner en riesgo la salud de operarios y pacientes.

1.1 Identificación de la Empresa

La clínica Odontología Familiar nace en 1991, como una clínica de servicio para los empleados de la Corporación Más x Menos (ahora Walmart). Inicia, con la atención por parte de un solo odontólogo, el Dr. Juan Carlos Quesada. Gracias a la calidad de sus procedimientos, al trato amigable con los pacientes y a la satisfacción de éstos, la clínica crece y logra establecerse como una práctica privada, ofreciendo sus servicios a cualquier persona que los requiera.

El doctor Quesada llegó a establecer dos consultorios, uno en el centro de San José y el otro en Guadalupe. Después de un tiempo determinado, decide mantener solo

el consultorio de San José debido a la centralización y a la facilidad de acceso por parte de sus pacientes.

Actualmente, la clínica se ubica del Automercado de San José Centro, 75 metros al Norte, en el Edificio Morano, segundo piso. Cuenta con un personal de 4 personas, 3 de las cuales son odontólogos y una asistente administrativa. Ofrece servicios desde cuidados odontológicos básicos hasta cirugía oral menor, extendiéndose a grupos de personas de cualquier edad. Tiene un enfoque especializado en el paciente, al que denominan “Odontología Sin Dolor”, con el fin de entregar la mejor calidad de sus tratamientos de una manera personalizada y que permita, que el paciente se sienta cómodo.

1.1.1 Misión

Somos una empresa que brinda un servicio de salud integral; contamos con terapias alternativas tales como: homeopatía, acupuntura, terapia neurofocal. En nuestras instalaciones encontrará un ambiente de respeto y calidez, donde será atendido por personal altamente calificado dispuesto a recibirlo siempre con una sonrisa.

1.1.2 Visión

Ser un centro de salud vanguardista en lo que respecta a terapias alternativas odontológicas, satisfaciendo las necesidades de cada uno de nuestros pacientes y sus familias, logrando reconocimiento a nivel nacional.

1.2 Justificación

La clínica Odontología Familiar emplea brocas dentales, en la mayoría de sus tratamientos. Sin embargo, no existe un control sobre el tiempo de uso, de cada broca, que permita estimar cuándo deben ser reemplazadas y desechadas. De existir este control, la administración podría calendarizar mejor, las fechas para realizar nuevos pedidos y esto a su vez, se traduce en un ahorro económico, ya que las brocas en inventario serían justo las necesarias para reemplazar las usadas.

Además, la comparación entre dos marcas de fabricantes diferentes proveerá información suficiente para que la administración pueda seleccionar confiablemente, las brocas que vayan a brindar las propiedades más óptimas a un costo balanceado.

Finalmente, la clínica ofrece un servicio enfocado y personalizado, denominado “Odontología Sin Dolor”. Anteriormente han detectado que, cuando las brocas están muy desgastadas y no funcionan convenientemente, los procedimientos generan incomodidad y dolor en el paciente, lo que los aleja de cumplir su ideal. Así, la sustitución a tiempo de las brocas va a reducir o incluso eliminar las molestias en los pacientes.

1.3 Objetivos

1.3.1 Objetivo General

- Cuantificar el desgaste mecánico de brocas esféricas de diamante para fresas odontológicas.

1.3.2 Objetivos Específicos

- Caracterizar el material de las brocas.
- Identificar los mecanismos de desgaste en las brocas y sus causas.
- Comparar la vida útil de las brocas de las fresas odontológicas de dos fabricantes distintos.

1.4 Alcances y Limitaciones

Existe una gran variedad de brocas en el mercado, que varían en material, tamaño y geometría. El interés de este trabajo no se centra en analizar cada uno de los tipos de brocas, sino que se ha elegido la más utilizada en los tratamientos más habituales. Así, este estudio pretende establecer una base teórico-práctica que permita analizar, el desgaste en las demás variaciones de geometría de esta pieza. Además, económicamente, representa un interés para la empresa, ya que al ser la más utilizada, es también la más reemplazada y la planificación del inventario, va a ser determinada en gran medida, por la vida útil de estas piezas específicas.

Por otro lado, a nivel técnico, ciertas variables en las muestras representan un inconveniente para realizar los ensayos y las mediciones propuestos. Los resultados que se obtienen son confiables, pero pueden no representar de manera exacta la realidad de lo que se está observando. Un primer caso, es la dureza de las piezas dentales extraídas, cuyas durezas varían ampliamente dependiendo de factores como la edad y el sexo de la persona, sus hábitos alimenticios, la presencia de reconstrucciones con otros materiales y el tejido, así como la profundidad, a la que se mida la dureza. Otro ejemplo, se presenta en la composición química de las brocas, ya que la superficie no es homogénea, sino que se trata de partículas dispersas, en una matriz metálica. Finalmente, el tamaño de las brocas dificulta el montaje de probetas para las pruebas, aunque no imposibilita el proceso.

2. Marco Teórico

2.1 Instrumentaria Dental

Un instrumento dental, definido por la UNE-EN 1639:2010, es un “instrumento especialmente diseñado para el uso en el ejercicio de la odontología. Puede ser accionado manualmente, a motor o de ambas maneras”. A su vez, un instrumento dental accionado a motor es “diseñado para ser accionado mediante una fuente de energía interna o externa de la que recibe la potencia necesaria para la función prevista”. Existen muchos tipos de estas herramientas, por lo que conviene abordarlos en los dos grupos mencionados anteriormente: los instrumentos manuales o simples y los instrumentos complejos o accionados a motor. [17]

Los instrumentos simples, que reciben también el nombre de estáticos, se conciben como extensiones de la mano del operador, sin necesidad de ser accionados por una fuente exterior. Por otro lado, los instrumentos complejos o dinámicos poseen cierta complejidad tecnológica y necesitan estar conectados a una fuente de energía para su óptimo funcionamiento. [17]

Dentro del grupo de instrumentos complejos, se encuentran los instrumentos rotatorios, que son los aparatos más utilizados para generar el desgaste tanto de los tejidos duros naturales como de los materiales sintéticos odontológicos. Conviene tener en perspectiva, la evolución de estos instrumentos, desde los mecanismos más sencillos que dieron paso a la gran complejidad actual que facilita su uso y funcionalidad.

2.1.1 Desarrollo Histórico de Equipos para Desgaste

Existen indicios de que la cultura maya y otras sociedades primitivas utilizaban arcos de perforación y otros instrumentos para realizar cavidades redondeadas en los dientes. A partir de mediados de los 1800, la operatoria dental vería muchos de los avances que dieron lugar a la disciplina tal como se conoce hoy. Se empleaban taladros de mano con puntas de acero, girando las palancas con los dedos (figura 1). Luego, se introdujo el uso de una fresa de dedo. Algunos taladros se modificaban mediante la incorporación de mangos flexibles entre las puntas y las palancas, lo que permitió el acceso a zonas más posteriores de la boca. El desarrollo de nuevos equipos mecánicos

a mediados de siglo, aceleró el avance en los equipos dentales; como ejemplo de estos, pueden mencionarse el taladro de Lewis, el taladro de Chevalier y el taladro de Merry. [14]

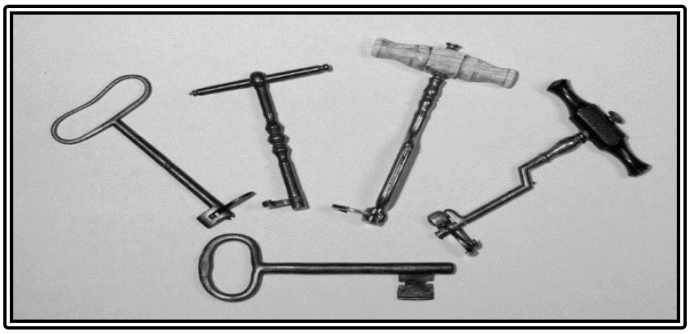


Figura 1. Taladros de mano usados en el siglo XIX.

Después del descubrimiento de la electricidad, a finales del siglo XVIII, los equipos odontológicos experimentaron un gran auge. El primer taladro eléctrico fue introducido en 1871, mismo año en que Morrison introdujo el primer motor de pedal dental, aunque para 1790, John Greenwood ya había fabricado el primer motor de pie (figura 2). Esto representó un incremento en la velocidad rotacional en comparación con los taladros operados con la mano. [14]

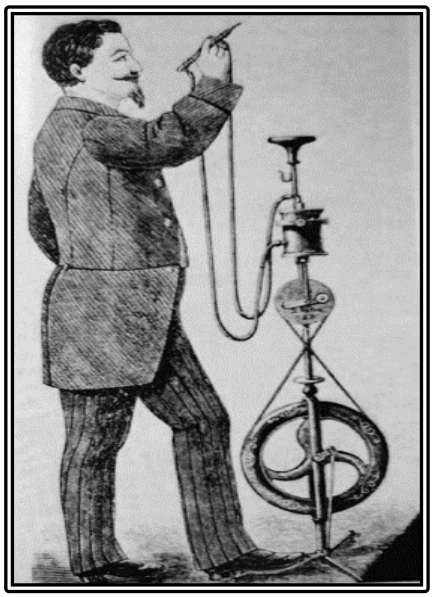


Figura 2. Taladro de John Greenwood.

Para 1891, la compañía S.S. White, introdujo las primeras fresas maquinadas, llamadas Revelation Burs ® (figura 3a). En este mismo año, Acheson descubrió un método para fabricar un abrasivo industrial compuesto de carburo de silicio, el cual patentó en 1893 bajo el nombre de Carborundum® (figura 3b). Por este tiempo, también se incluyeron instrumentos de diamante y corundo para preparación de cavidades. [14]

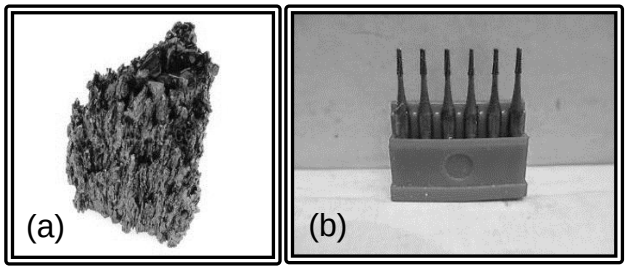


Figura 3. (a) Carborundum®; (b) Revelation Burs®.

A pesar de la introducción de los equipos eléctricos, los taladros de pedal aún se utilizaban mayoritariamente hasta aproximadamente 1915. El siglo pasado, vió la puesta en funcionamiento de muchos de los aparatos que son predecesores de las piezas de mano usadas en la actualidad, como lo es el Dentalair ® de Norlen en 1948 (figura 4a), la pieza de mano con turbina de aire de Walsh en 1949, su variante con turbina de agua en 1952 y la pieza de mano de alta velocidad de Page, en 1955. [14]

Sin embargo, no sería hasta 1957 con la introducción del Airotor ® de John Borden (figura 4b), que la práctica dental sería revolucionada. Esta era operada por aire y alcanzaba velocidades de hasta 200 000 rpm. A partir de este diseño, evolucionaron los equipos más modernos. En 1947, se había desarrollado la primera fresa de carburo, pero no sería sino hasta la presentación del Airotor®, que estas desplegarían su máximo potencial. [14]

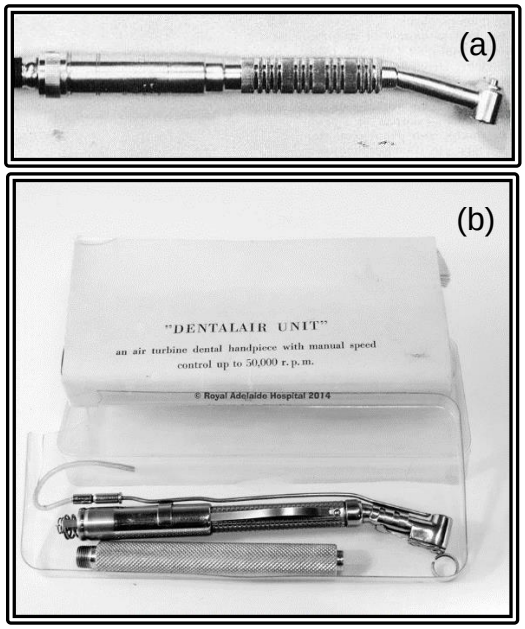


Figura 4. (a) Dentalair®; (b) Airotor®.

Hoy, el avance de estos equipos es notorio; las principales ventajas que presentan son el bajo ruido de operación, buena concentricidad y muy alto torque, aunque esto involucre un alto costo de inversión inicial y de mantenimiento.

2.1.2 Instrumentos Rotatorios

El enfoque de este trabajo se centra en instrumentos que ocasionan el desgaste en las piezas dentales debido a su movimiento rotatorio. Cada uno de los elementos que lo componen están regulados por normas UNE ISO. A continuación, se describen de manera general las partes que los constituyen.

2.1.2.1 Adaptadores

Estos componentes permiten la conexión del instrumento con mangueras para aire comprimido, agua, iluminación o cámaras, o entre piezas del mismo instrumento, como los motores y las piezas de mano. Su función y, por ende, su diseño, varían grandemente en el mercado, dependiendo de la empresa que los fabrica. Lo que caracteriza a cada adaptador es el número de canales que contiene en su interior y su

diámetro, los cuales alojan las mangueras para las funciones mencionadas (figura 5). [17]

Figura 5. Adaptadores de pieza de mano.

2.1.2.2 Elementos Motrices

Son los que realizan el movimiento rotatorio o giratorio (figura 6). Los micromotores (llamados así por sus dimensiones reducidas), permiten una disminución en el peso y el volumen del aparato. Esto facilita realizar maniobras más precisas y complejas con mayor comodidad. Se distinguen dos clases: los potenciados por energía eléctrica o los movidos por aire comprimido. Los primeros operan con un voltaje bajo, con el fin de prevenir accidentes eléctricos. Por esta razón, trabajan solo a baja velocidad, las que alcanzan entre 30 000 y 40 000 r.p.m. Por otra parte, el otro tipo de elementos motrices puede actuar tanto a bajas como a altas velocidades, gracias al movimiento generado por las turbinas accionadas por un flujo de aire a presión. Se encuentran actualmente en el mercado motores que alcanzan las 300 000 o 400 000 r.p.m. Esto, sin embargo, conlleva la necesidad de emplear un sistema de enfriamiento constante y otro de aspiración. [17]

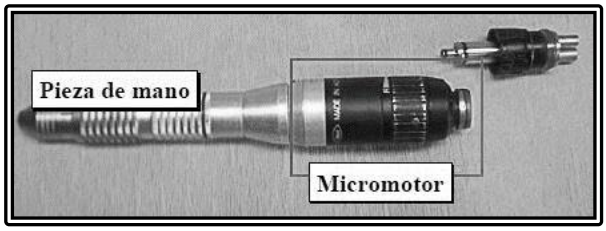


Figura 6. Micromotor de una pieza de mano.

2.1.2.3 Piezas de mano

Son los elementos que transmiten el movimiento rotatorio desde donde se origina (motor) hasta la pieza activa (fresa). Se fabrican normalmente de acero o titanio. Pueden distinguirse dos tipos de piezas de mano: las rectas y las contra-anguladas. Las primeras son una prolongación longitudinal del eje del aparato (figura 7a). Las contra-anguladas, poseen una angulación obtusa en la parte media y un ángulo recto en la cabeza (figura 7b). Esta disposición permite el fácil acceso de la pieza a cualquier zona de la cavidad bucal. [17]

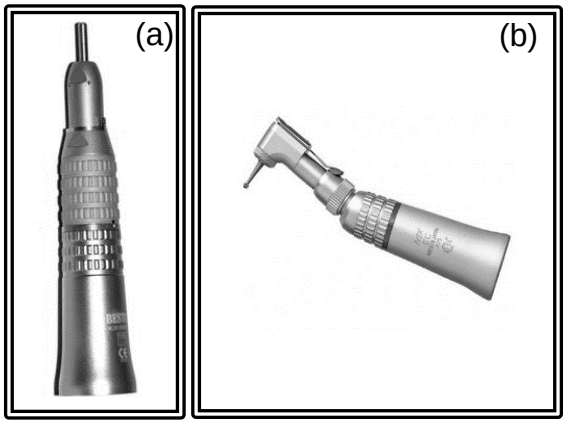


Figura 7. (a) Pieza de mano recta.
(b) Pieza de mano contra-angulada.

2.1.2.4 Elementos Activos

Son los que producen el corte, el desgaste y el pulido en los materiales dentales. Se les conoce comúnmente como fresas o brocas. Estas piezas varían mucho en su composición y morfología. La elección de estos elementos depende de la operación que se va a desarrollar y su fin en el procedimiento odontológico. La forma está definida en

la norma, como “figura geométrica de la envolvente de rotación descrito por la pieza de trabajo de un instrumento rotatorio, durante su rotación axial”. Además, de la geometría, es importante tomar en cuenta que cada una presenta diámetros y dimensiones distintos. Las formas más comúnmente utilizadas se describen a continuación (figura 8). [17]

- Esférica o redonda: se utiliza para la remoción de caries y la generación de cavidades en piezas dentales, que luego serán calzadas con resina o amalgama. El desgaste que van a presentar se localiza mayoritariamente en su extremo superior y algunas veces, se pueden desgastar desde los lados, cuando se genera una cavidad en varias direcciones.
- Cilíndrica: estas pueden ser activas en su extremo, cuando se busca aumentar la profundidad de una cavidad o aplanar el fondo de esta, o pueden ser utilizadas lateralmente, cuando se requiere ampliar la cavidad hacia los lados.
- Cónica invertida: a diferencia de la cónica, su base está alejada del eje. Esta broca se usa para generar una cavidad cuya forma de cono invertido genera una retención de las paredes del diente.
- Troncocónica invertida: describe una forma de cono truncado. La longitud de su cabeza y del diámetro son aproximadamente iguales. Cumple funciones similares a la cónica invertida.

Además de estas figuras, también pueden conseguirse en el mercado otras más específicas, como las anilladas, en forma de llama, de doble cono, en forma de pera, ovoides, de barril, entre muchas otras (figura 8).

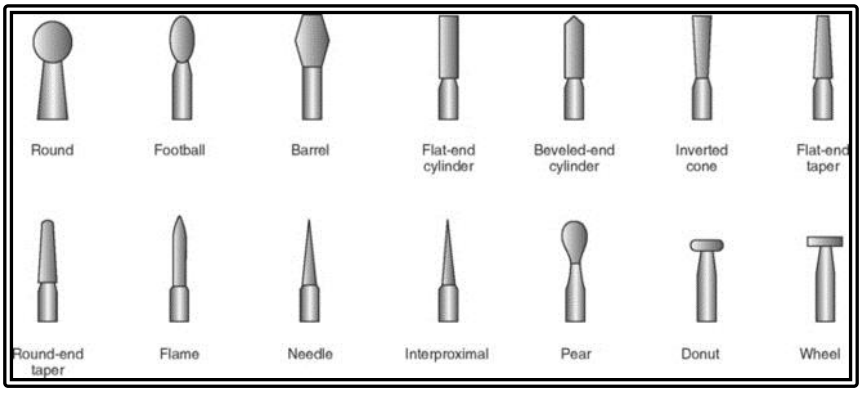


Figura 8. Formas de brocas dentales.

2.1.3 Normativa ISO

La fabricación y el dimensionamiento de las brocas de distintos materiales y geometrías son regulados por una serie de normas ISO. A continuación, se mencionan las relacionadas a los elementos activos rotatorios en general y a las brocas de diamante específicamente:

- ISO 1797-1:2011 Shanks for rotary instruments – Part 1: Shanks made of metals
- ISO 2157:2016 Nominal diameters and designation code numbers for rotary instruments
- ISO 6360-2:2004 Number coding system for rotary instruments – Part 2: Shapes
- ISO 6360-4:2004 Number coding system for rotary instruments – Part 4: Specific characteristics of diamond instruments

En conjunto, estas normas identifican cada tipo de broca, como se mostrará en la sección de Resultados y Análisis.

2.2 Estructura del Diente

Los dientes deben poseer ciertas características químicas que lo protejan y mantengan estable, ante las muchas sustancias que la boca alberga y otras mecánicas, que les permitan realizar la función de trituración de la comida, sin que se deterioren rápidamente. A su vez, estas propiedades mecánicas van a influir directamente en el desgaste de las brocas. La estructura del diente está compuesta por 4 tejidos diferentes:

el esmalte, la dentina, la pulpa y el cemento, cada uno de los cuales, posee propiedades distintas pero que, en conjunto, permiten el desarrollo y desempeño ideal, de las piezas dentales. Estos atributos varían en un amplio rango de acuerdo a la edad, hábitos alimenticios, rasgos hereditarios, tipo de pieza dental, inclusive la localización en la misma pieza, y un sin número de factores tanto médicos como sociales. Sin embargo, puede realizarse una descripción general de sus cualidades.

2.2.1 Esmalte

El esmalte dental es el tejido que se encuentra en interacción con el entorno, por lo que es el más duro y altamente mineralizado de todo el cuerpo humano. Gracias a esto, protege los tejidos más internos del diente. Está compuesto de aproximadamente un 96% de hidroxiapatita (HA), un 3% de agua y una 1% de proteína. Los ejes de HA, se combinan en estructuras delgadas y largas de pocos milímetros de diámetro, similares a fibras, las cuales se conocen como prismas. Estos prismas se unen mediante una capa muy delgada de proteína (figura 9). [13]

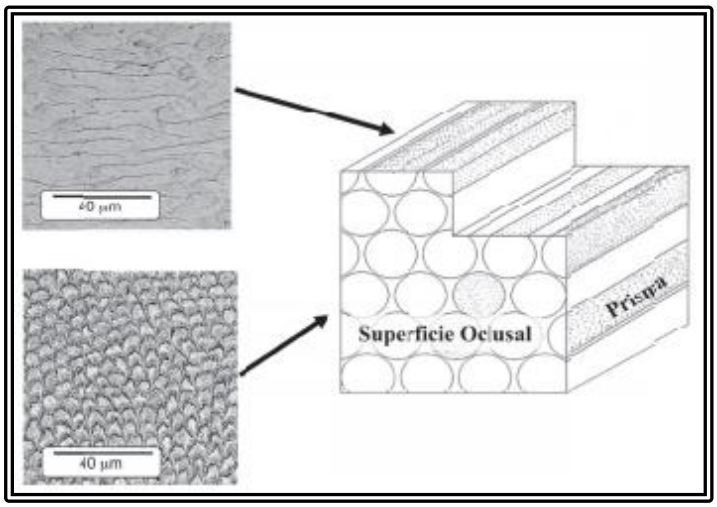


Figura 9. Disposición de la HA en prismas alargados.

El módulo de elasticidad, la dureza, la tenacidad y la fragilidad del esmalte han sido estudiados anteriormente y se han registrado valores similares a los del circonio, el oro, la plata y el vidrio de borosilicato, que son materiales muy resistentes a las cargas a

las que se someten. Por ejemplo, el módulo de elasticidad se ha calculado entre 70 y 120 GPa y su dureza, entre 3 y 6 GPa. [3] Esto indica que el esmalte posee muy buenas propiedades mecánicas, a pesar de ser compuesto en su mayoría por material inorgánico. Sin embargo, en una misma pieza dental, los prismas de HA pueden orientarse de manera distinta, variando las propiedades del esmalte y volviéndolo un material anisotrópico. Algunos autores, han nombrado las zonas de orientación similar en los prismas, siendo que cuando estos se orientan paralelo al observador, se conoce como parazona, y cuando, se encuentran dispuestos perpendicularmente, se denomina diazona (figura 10). [13]

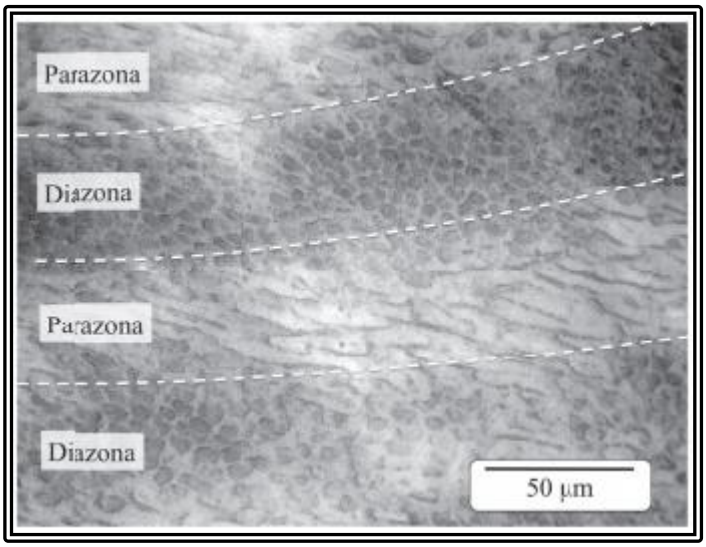


Figura 10. Orientación de los prismas del esmalte en una pieza dental.

2.2.2 Dentina

Es el componente que se encuentra en mayor proporción en los dientes. La caracterización de este tejido, ha permitido la creación de nuevos materiales restaurativos que puedan sustituirla y cumplir sus funciones. Su composición química consiste de 50% de cristales de HA, 30% de material orgánico (mayoritariamente colágeno tipo I) y 20% de un fluido similar al plasma de la sangre. La dentina también es anisotrópica, ya que está compuesta por túbulos dentinarios que se orientan en varios

sentidos. Los túbulos consisten en cristales de HA que engloban fluido dentinario (figura 11).

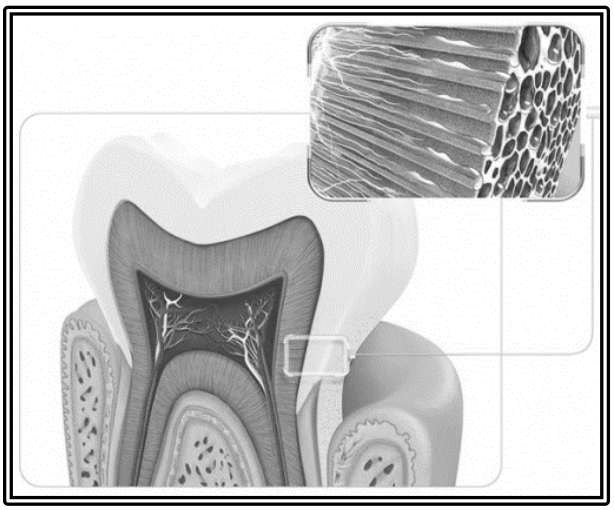


Figura 11. Túbulos dentinarios con fluido dentinario.

Se han llevado a cabo numerosos estudios para determinar las propiedades mecánicas de la dentina, aunque los resultados varían mucho entre sí, según el método de ensayo usado y de las características de cada paciente. Muchos estudios fueron realizados en esta dirección, variando el método de ensayo y obteniéndose resultados muy variados. A pesar de esto, se ha concluido que la mayoría de propiedades de interés, aumenta cuando la carga es aplicada perpendicularmente al eje de los túbulos; los esfuerzos generados son de tensión. Cuando la carga es aplicada paralelamente, se producen esfuerzos de cizalla, los que se propagan más rápidamente. [4]

La dureza se ha medido mediante microindentacción Vickers y se han obtenido valores que varían entre 250-800 MPa, dependiendo de la ubicación de la indentación con respecto a los demás tejidos dentales. La dentina peritubular, es varias veces más dura que la intertubular, debido a que su contenido mineral es casi un 95% en volumen, en contraste con el 30% que posee la dentina intertubular. En cuanto al módulo de elasticidad, se puede decir que la dentina es relativamente rígida, registrando valores de 10-20 GPa. La elasticidad de la dentina es de gran importancia, ya que compensa la rigidez del esmalte y permite una mejor distribución de esfuerzos debidos a la actividad

masticatoria. Esta varía de acuerdo al contenido de material orgánico y agua que se encuentra en la estructura dentinaria. [4]

2.2.3 Pulpa

La pulpa es un tejido vivo, altamente inervado y vascularizado. Está formada por un 75% de agua y un 25% de material orgánico. [9] Es el tejido conectivo que mantiene y forma la dentina durante toda la vida, aunque con la edad va disminuyendo su volumen. [2] La pulpa es el tejido más blando del diente, lo que supone que sus propiedades mecánicas pueden subestimarse al ser tan bajas. Su función sensorial se limita a transmitir la sensación de dolor por medio de las fibras nerviosas. [16]

Su estructura consiste de 4 zonas (figura 12): [8]

- Capa de odontoblastos: se presenta debajo de la dentina
- Zona acelular o de Weil: posee pocas células. Se localizan los vasculares que alimentan a los odontobalstos y a la dentina.
- Zona rica en células: es donde se encuentran las células madre de la pulpa.
- Pulpa central: presenta muchos vasos sanguíneos y nervios, además de distintos tipos celulares.

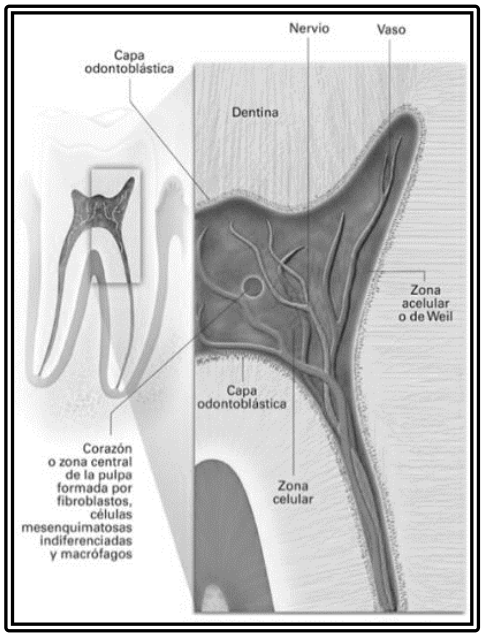


Figura 12. Zonas de la pulpa dental.

2.2.4 Cemento

Es un tipo de tejido conectivo calcificado que recubre la raíz del diente y cumple una función de anclaje de la pieza dental al hueso alveolar de la encía (figura 13). No está vascularizado, ni inervado. [10] Es menos duro, que la dentina debido a que la cantidad de mineral presente es menor. Su composición química consiste aproximadamente de un 65% de material inorgánico, presente en forma de cristales de HA y fosfato de calcio en estado amorfo; material orgánico en un 23% compuesto de colágeno; y un 12% de agua. [1] Ribeiro, Alexandre y Hildebrando (2004) registran valores de microdureza Vickers en el cemento radicular, promediando 19,70 HVN.

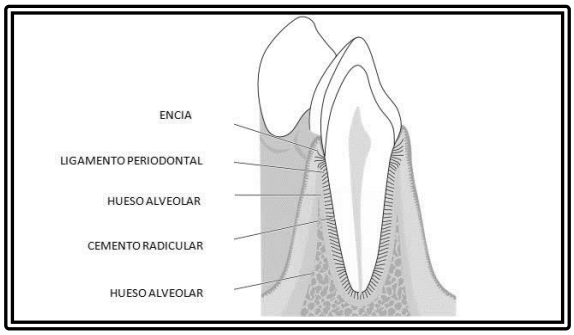


Figura 13. Cemento reticular en el diente.

2.3 Mecanismos de Desgaste

Los procedimientos odontológicos hacen uso de distintos mecanismos de modificación de la superficie dentaria para tratar los diversos problemas encontrados en los pacientes. La gran variedad de equipos desarrollados se diseña de acuerdo a la función que van a ejercer y a la acción mecánica, que van a ejecutar durante su funcionamiento. Muchas veces, estos tratamientos requieren la exposición de la estructura interna del diente para realizar restauraciones, eliminar caries o extraer piezas. Para ello, es necesario retirar el material que permite al acceso a esas zonas internas, dando atención a la comodidad del paciente, por lo cual se debe ir desgastando progresivamente la superficie. Los instrumentos van a sufrir desgaste de igual manera.

Existen varios mecanismos de desgaste que a su vez que son empleados por una variedad de instrumentos dentales.

El desgaste se define como la remoción de material de un elemento causado por el movimiento relativo de este respecto a otro con el que se encuentra en contacto. El material removido puede adherirse a la otra superficie, permanecer atrapado entre ambas superficies o ser expulsado de entre estas. La fuerza de fricción entre las áreas en contacto está asociada a la evolución del desgaste, aunque su influencia no ha sido muy bien entendida. El proceso consiste en un arranque gradual de partículas pequeñas cuyas dimensiones varían ampliamente. El desgaste puede ser ocasionado por fenómenos mecánicos (adhesivo, abrasivo y erosivo) o fenómenos químicos. [5]

2.3.1 Desgaste Adhesivo

Se presenta debido al deslizamiento de una superficie más o menos lisa contra otra de baja rugosidad también. Los puntos de contacto entre los planos que se deslizan pueden adherirse entre sí en un instante y debido al movimiento, ser fragmentados posteriormente. El desgaste, que se presenta mayoritariamente en el material más blando, va a depender del tiempo, y generalmente, sucede lentamente. Además, la presión entre ambas superficies también ejercerá influencia sobre la velocidad del proceso, así como la presencia de fuerzas de impacto o choque de una superficie contra la otra. [7] [5]

Para estimar el volumen del desgaste por adhesión, se utiliza la relación observada por Holm (1946) y Archard (1953), la cual viene expresada por:

$$V = \frac{kWx}{H} \qquad \textit{(Ecuación 1)}$$

donde el volumen del desgaste V, es proporcional a la carga aplicada W, a la distancia de desplazamiento x, e inversamente proporcional a la dureza de la superficie desgastada (más blanda) H, siendo k el coeficiente de desgaste de los materiales en contacto. Aunque la Ecuación 1, es independiente a la velocidad del deslizamiento, en algunos casos se exhibe una influencia en el desgaste a velocidades críticas. [5]

Normalmente, este mecanismo se emplea en el pulido final de piezas dentales, ya que alisa la superficie dental a la vez que le proporciona su lustre característico.

2.3.2 Desgaste Abrasivo

Producido por el deslizamiento de partículas duras o de una superficie rugosa sobre una superficie más lisa. El desgaste se produce por deformación plástica y fractura en el área de contacto. Este fenómeno se presenta en dos casos típicos. En el primer caso, una superficie dura roza y produce abrasión en la más blanda (figura 14a), y en el segundo caso se presentan pequeñas partículas libres (algunas veces generadas inicialmente por desgaste adhesivo), las cuales se comportan como material duro que causa abrasión en una o las dos superficies (figura 14b). [5]

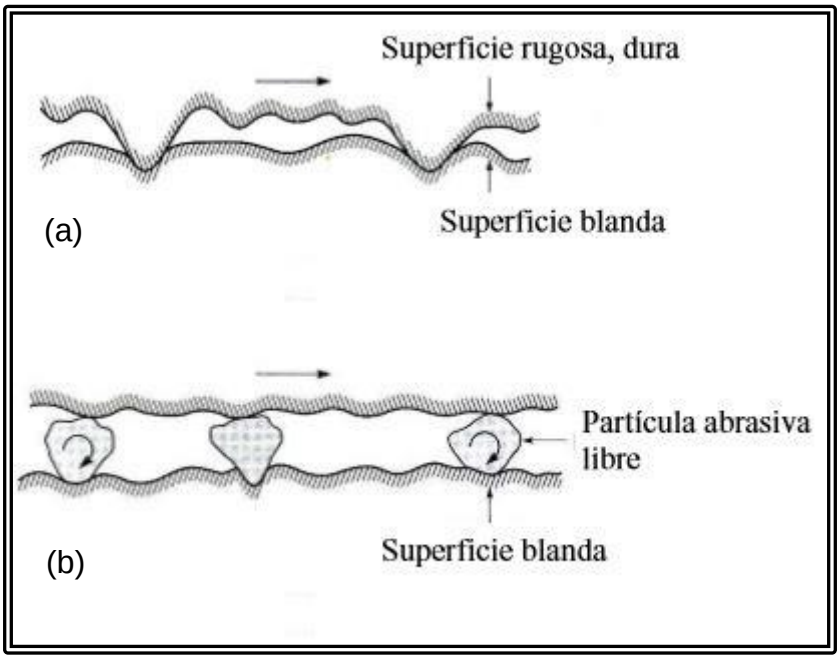


Figura 14. (a) Desgaste abrasivo entre una superficie dura y otra blanda y (b) desgaste abrasivo generado por partículas libres entre las superficies.

De manera similar a la Ecuación 1, el volumen desgastado por abrasión V, se puede calcular mediante la expresión:

$$V = \frac{k_{abr} W s}{H} \qquad \textit{(Ecuación 2)}$$

donde k_{abr} es el coeficiente de desgaste adimensional, W es la carga aplicada, s es la longitud de la traza de abrasión y H es la dureza del material más blando. [5]

El caso más característico de este mecanismo, es el empleo de fresas en los instrumentos rotatorios. Las brocas, son de un material muy duro y abrasivo, que genera el desgaste en los materiales más blandos presentes en el diente. También se utiliza en operaciones de desbastado y tallado. En el primer procedimiento, se busca eliminar residuos, rebabas, imperfecciones groseras y cualquier irregularidad de la superficie dental; el segundo proceso consiste en dar forma al tejido del diente, con el fin de mejorar su morfología final o para la aplicación posterior de otras técnicas, como restauración y relleno con resinas.

2.3.3 Desgaste Erosivo

Se exhibe al chocar partículas pequeñas a alta velocidad contra una superficie, generando una fuerza de impacto muy grande (figura 15). La erosión puede generarse por el transporte en un vehículo líquido (usualmente aire o agua), de las partículas sólidas pequeñas. La velocidad de las partículas, el ángulo en que impactan y su tamaño determinan la energía cinética, que a su vez permite estimar la fuerza de impacto. [5]

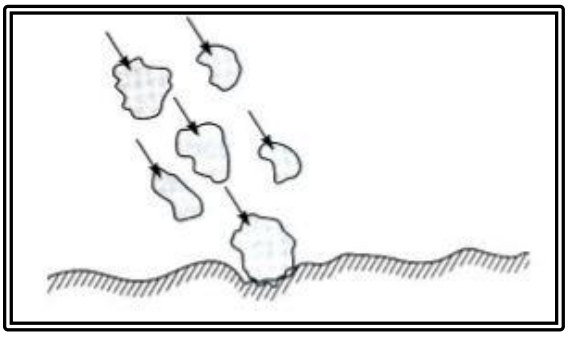


Figura 15. Desgaste generado por erosión.

En materiales dúctiles, el desgaste se da por deformación plástica; en los frágiles, el material se desgasta por la formación de grietas que se originan en el punto de impacto de la partícula dura y se cruzan entre sí. [5]

El volumen desgastado por erosión se describe en la Ecuación 3:

$$V = KA(\alpha)v^n M^3 \qquad \textit{(Ecuación 3)}$$

donde V, representa el volumen de desgaste, K es una constante empírica, A es una función del ángulo de impacto α (figura 16), v es la velocidad de las partículas, n depende del material (2-2.5 para metales y 2.5-3 para cerámicas) y M es el tamaño de partícula. [7]

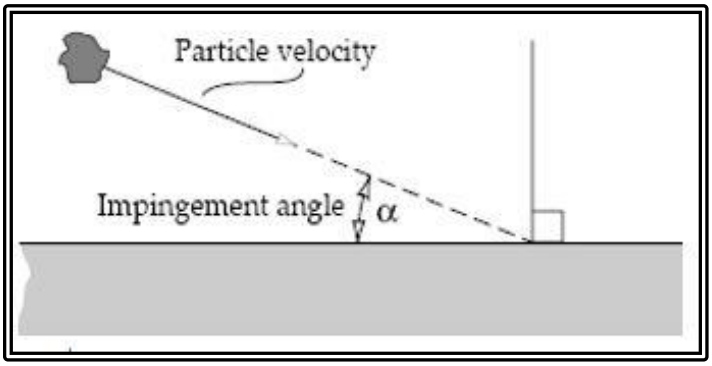


Figura 16. Ángulo de impacto en partículas erosivas.

2.3.4 Desgaste Químico

Se presenta cuando el deterioro ocurre en un ambiente corrosivo; por esta razón se le denomina también desgaste corrosivo. Ante la presencia del medio corrosivo, sea líquido o gaseoso, el material expuesto reacciona químicamente y se forma una capa de óxidos en la superficie del material, la que podría detener el avance de la corrosión (pasivación). Sin embargo, debido al deslizamiento relativo de las zonas en contacto, esta capa se fracciona, lo que permite que el ataque continúe (figura 17). Las partículas desprendidas son muy pequeñas y actúan como agentes abrasivos, lo que ocasiona un daño mayor. [5]

Ningún equipo odontológico, utiliza este tipo de desgaste para llevar a cabo sus operaciones; sin embargo, se puede generar este fenómeno en los dientes y en los aparatos debido a la presencia de sustancias con diferentes niveles de acidez, como jugo gástrico, saliva, alimentos y bebidas, entre otros. Los equipos también pueden experimentar este tipo de desgaste al ser esterilizados con soluciones con un pH mayor a 11. [6]

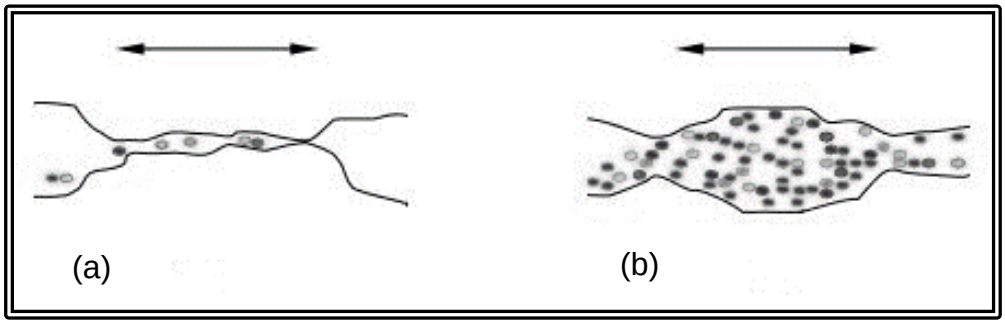


Figura 17. (a) Inicio del desgaste. (b) Avance del desgaste corrosivo.

3. Metodología

Para alcanzar los objetivos propuestos, se llevaron a cabo una serie de actividades sistemáticamente. La tabla 1, resume los aspectos más relevantes de estas actividades, que posteriormente son ampliados.

Tabla 1. Plan de actividades según los objetivos.

Plan de Acción			
Objetivo General: Cuantificar el desgaste mecánico de brocas esféricas de diamante para fresas odontológicas.			
Objetivo Específico	Productos	Actividades	Responsable por actividad
OE1 Caracterizar el material de las brocas.	Propiedades y características del material.	Medición de durezas.	Juan José Alvarado Asistente en laboratorio
		Determinación de composición química de las brocas mediante EDS en el Laboratorio de Microscopía.	Juan José Alvarado Héctor Agüero
OE2 Identificar los mecanismos de desgaste en las brocas y sus causas.	Factores que influyen en el desgaste de las brocas	Investigación bibliográfica sobre tribología en los materiales.	Juan José Alvarado
		Observar y describir los distintos procesos en los que se utilizan las brocas, mediante investigación bibliográfica y observaciones presenciales.	Juan José Alvarado Juan Carlos Quesada
OE3		Tabular el tiempo de uso y la pérdida	Juan José Alvarado

Comparar la vida útil de las brocas de las fresas odontológicas de dos fabricantes distintos.	Vida útil de las brocas, fabricante recomendado, programación para reemplazo	de masa de cada broca.	Dra. Pamela Herrera Asistente en CEQIATEC Héctor Agüero
		Calcular el costo económico de reemplazar las brocas en un lapso igual al período de experimentación.	Juan José Alvarado
	Informe final	Redactar informe final	Juan José Alvarado

Para el objetivo específico 1, se determinó la composición de los materiales de las brocas y se consideraron algunas propiedades pertinentes para el estudio que se va a realizar. Para ello se utilizó el microscopio electrónico de barrido TM-1000 marca Hitachi, del Laboratorio de Microscopía del TEC y se realizaron medidas de dureza con el durómetro Vickers marca Mitutoyo del Centro de Investigación y Extensión en Materiales. Se tomaron como referencia documentos recientes sobre las propiedades de estos componentes y los catálogos de los proveedores que se encuentran en línea.

Para el objetivo específico 2, se realizaron varias observaciones presenciales de los procedimientos odontológicos, en los que se utilizan las brocas. Esta actividad se complementa con la recopilación de varios documentos que describen estos procedimientos. Además, se determinó de qué manera, se produce el desgaste en las brocas y cuáles agentes los causan. Investigaciones recientes similares fueron consultadas como fundamento para el análisis. También se referenciaron los parámetros de operación del equipo y se aludió a la esterilización antes del uso de cada broca.

Finalmente, para el objetivo específico 3, se realizaron imágenes microscópicas de las superficies de las brocas desgastadas. También se documentó el cambio de dimensiones, masas y presencia de agentes extraños. De acuerdo al valor unitario de las brocas de los distintos proveedores, al cronograma de pedidos y al mejor rendimiento observado durante el experimento, se determinó la broca más adecuada para los

procedimientos clínicos, con un adecuado balance económico y con una vida útil prolongada.

3.1 Experimento

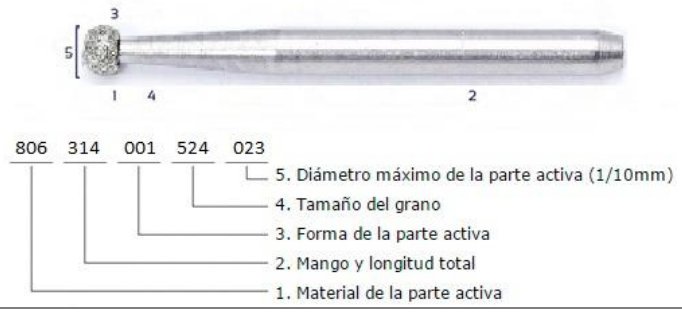
Clínicamente, las condiciones que afectan el uso y desgaste de una broca odontológica son muy variadas y difícilmente, pueden reportarse con exactitud. Por ejemplo, pueden mencionarse los siguientes factores:

- Edad del paciente: en general, conforme aumenta la edad, los tejidos del diente se vuelven más blandos.
- Hábitos del paciente: las sustancias que ingiere el paciente pueden ablandar los tejidos o acumularse entre los dientes y producir caries, sarro o cálculo dental, con propiedades tribológicas distintas.
- Material del diente: los dientes pueden contener distintos materiales con durezas muy diferentes, ya sean de tejido natural o materiales para reconstrucción, como resinas poliméricas, amalgamas metálicas o combinaciones de estos.

Debido a estas condiciones y a los alcances de este trabajo, no se empleó la metodología en casos clínicos. Para cuantificar y controlar las variables, sin perder confiabilidad, de los resultados, se diseñó un experimento a ser ejecutado en laboratorio, que simulara lo más fielmente posible las condiciones en pacientes clínicos.

El experimento empleó 3 brocas esféricas de dos fabricantes distintos: Jota y Coltène. Las brocas utilizadas se identifican con el número ISO 806314001524023, siendo cada uno de los parámetros especificados en la tabla 2.

Tabla 2. Especificación de la designación ISO para las brocas utilizadas.



1	2	3	4	5
Polvo de diamante	Turbina FG, longitud estándar 19mm, Ø 1,6mm	Forma esférica	Grano estándar 64-126 µm	Medida 2,3mm

Se prepararon cavidades en piezas dentales extraídas con cada broca durante 30min. Las piezas dentales no contenían ningún material de reconstrucción. En el tiempo 0 se tomaron micrografías para observar la geometría. Se midió la masa de cada broca. Este procedimiento se repetiría cada 30min de uso para observar el cambio en sus dimensiones y cuantificar la reducción de masa. Las brocas se secaron durante 30min a 110ºC antes de ser pesadas, con el fin de extraer la humedad y obtener una medida confiable de la masa.

Antes de cada uso, cada broca se esterilizó con vapor a 200°C durante 45min. Normalmente, la velocidad de rotación varía de acuerdo al usuario, dependiendo de la dureza del material que se esté trabajando. Sin embargo, para mantener la igualdad de condiciones en el experimento, se trabajó a un promedio de 35000 r.p.m. También, se trabajó con salida de agua, por la parte superior de la pieza de mano, como enfriamiento y mecanismo de arrastre de partículas desprendidas. Todas estas condiciones corresponden a las utilizadas en procedimientos con pacientes.

4. Resultados y Análisis

A continuación, se presentan los resultados de las mediciones realizadas, además de un análisis que aclara cada uno de los fenómenos observados. En primer lugar, para facilidad de identificación, las brocas Coltène se nombraron C1, C2 y C3; las brocas Jota se nombraron J1, J2 y J3.

Un análisis químico de cada broca se llevó a cabo mediante EDS. Los porcentajes mostrados, no representan con exactitud la cantidad de cada elemento, ya que el análisis, se realiza sobre un volumen determinado, que no solo contiene la sección de la pieza en estudio, sino que también contempla el portamuestras y el sistema de montaje, por lo que registra también la composición de estos componentes. Aunque los resultados son una conclusión cualitativa y no cuantitativa de las muestras, esto permite identificar apropiadamente los elementos en el material y brinda una noción general sobre la proporción en la que se encuentran.

Existen trazas de elementos que no son de interés para esta investigación, como el silicio en C1 (figura 18) y el flúor en C2 (figura 19). Este mismo análisis químico se realizó en C3, la cual contiene, en mayor o menor proporción, los mismos elementos (figura 20).

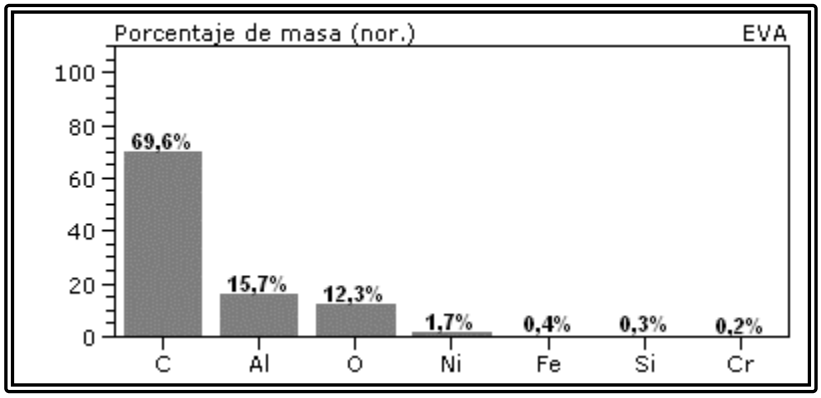


Figura 18. Elementos presentes en la broca C1 determinados mediante EDS.

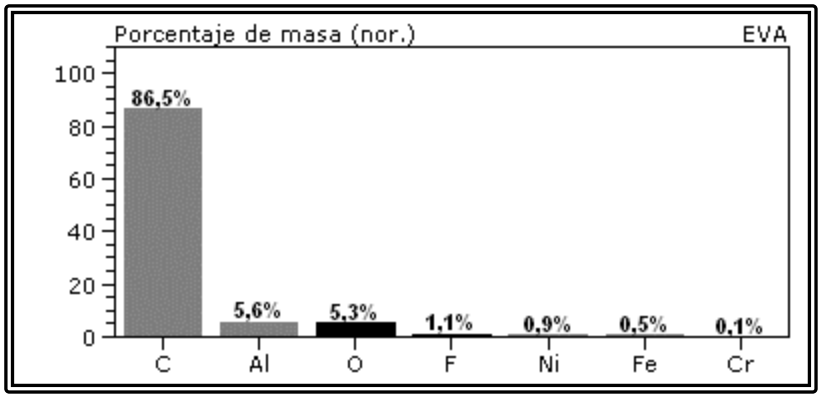


Figura 19. Elementos presentes en la broca C2 determinados mediante EDS.

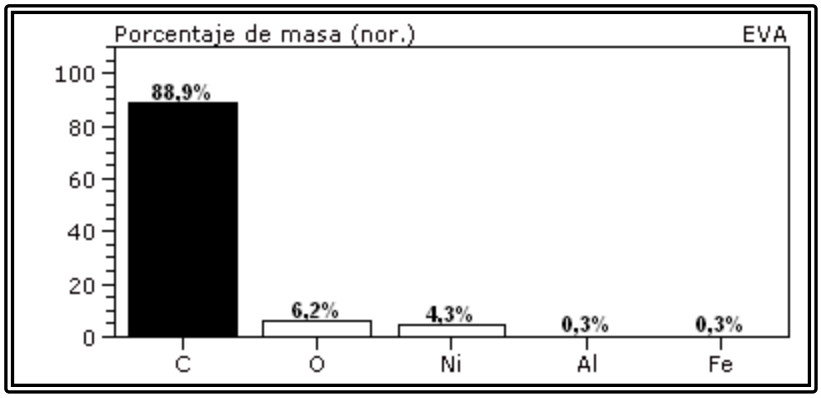


Figura 20. Elementos presentes en la broca C3 determinados mediante EDS.

El mismo estudio químico se realizó en J1 (figura 21), J2 (figura 22) y J3 (figura 23), las cuales presentaron resultados similares a los en C1, C2 y C3. Se advierte una alta proporción de C correspondiente a la presencia de cristales de diamante. Los cristales de diamante se depositan sobre una matriz de níquel o níquel-cromo en el proceso de manufactura mediante electrodeposición, lo que justifica la presencia de estos elementos en medianas proporciones. Además, las brocas son fabricadas en acero inoxidable posiblemente aleado con estos elementos para conferirle ciertas propiedades particulares; por ejemplo, el níquel mejora la resistencia a la corrosión e incrementa la tenacidad y el cromo aumenta la resistencia al desgaste y en caliente y proporciona inoxidabilidad. [15] [11]

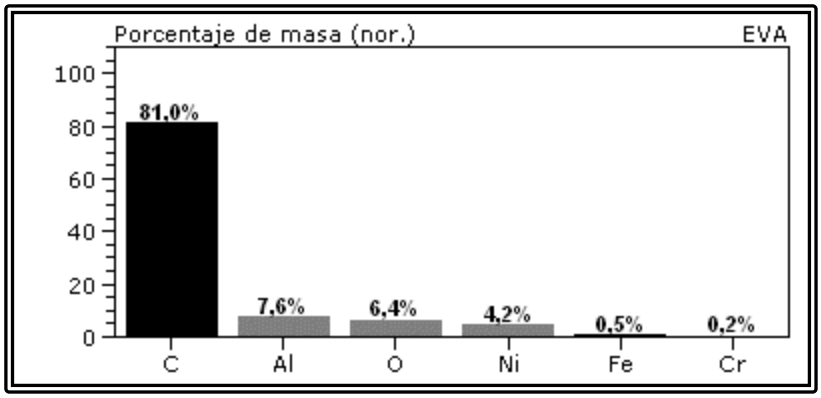


Figura 21. Elementos presentes en la broca J1 determinados mediante EDS.

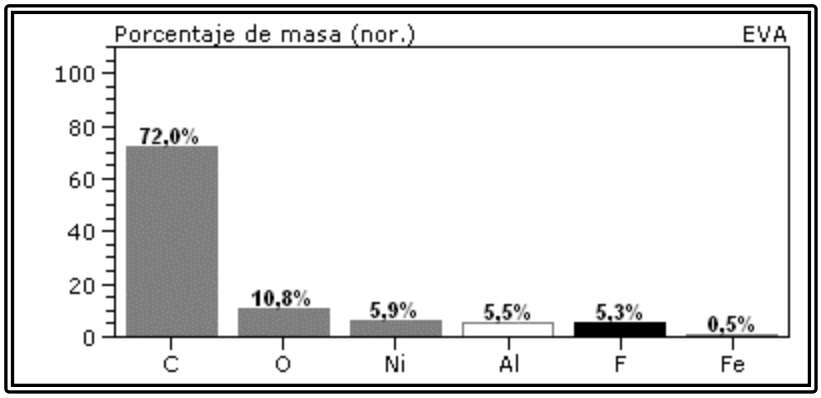


Figura 22. Elementos presentes en la broca J2 determinados mediante EDS.

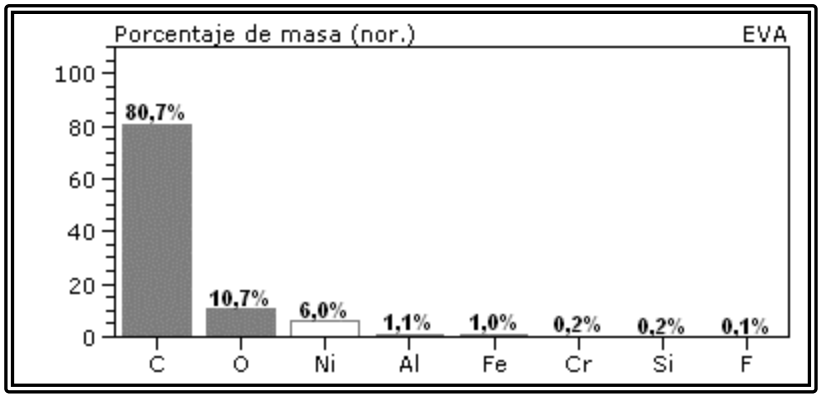


Figura 23. Elementos presentes en la broca J3 determinados mediante EDS.

Como valores iniciales, se midió la masa de cada broca en una balanza analítica y se aproximaron, los diámetros en el eje *x* y en el eje *y* de la esfera, sin ser usadas

(0min) (tabla 3). Se midieron de nuevo los valores de la masa y la longitud de los diámetros en ambos ejes después de 30min de uso (tabla 4). Se observó un desgaste avanzado en las brocas, por lo que la última medida se realizó a 45min para que los mecanismos asociados fueran observables con facilidad y no se afectara la superficie en estudio de manera excesiva. Los resultados de las medidas de las diagonales tomadas a 45min se presentan en la tabla 5.

Tabla 3. Valores de la masa y los diámetros de las brocas, 0min.

Broca	Masa (±0.001) g	Diámetro (±0.01) x10^{-3}m	
		x	*y*
C1	0,2823	2,10	1,91
C2	0,2820	2,08	1,85
C3	0,2820	2,10	1,86
J1	0,2725	2,11	1,98
J2	0,2736	2,14	1,94
J3	0,2740	2,08	2,11

Tabla 4. Valores de la masa y los diámetros de las brocas,30min.

Broca	Masa (±0.001) g	Diámetro (±0.01) x10^{-3}m	
		x	y
C1	0,2827	1,97	1,80
C2	0,2819	1,97	1,84
C3	0,2820	2,10	1,88
J1	0,2725	1,95	1,83
J2	0,2734	1,93	1,83
J3	0,2737	1,95	1,71

Tabla 5. Valores de la masa y los diámetros de las brocas, 45min.

Broca	Masa (±0.001) g	Diámetro (±0.01) x10^{-3}m	
		x	y
C1	0,2833	1,97	1,80
C2	0,2822	1,95	1,80
C3	0,2826	1,87	1,68
J1	0,2727	1,98	1,81
J2	0,2734	1,96	1,82
J3	0,2741	1,95	1,80

De las tablas anteriores, se advierte que las brocas C tienen una masa levemente mayor que las brocas J. Respecto a los cambios netos de masa, en el caso de las brocas C, se presentan cambios de entre $2x10^{-4}$ y $1x10^{-3}$ g, en contraste con el cambio neto observado en las brocas J, de entre $1x10^{-4}$ y $1x10^{-3}$ g; en otras palabras, el cambio de masa neto es mayor en las brocas C por hasta un orden de magnitud. Si se observa el tamaño de los cristales en cada marca de broca en la figura 24, se percibe que estos son más grandes en las brocas C, por lo que el espacio libre entre estos es más grande. Esto explica la tendencia a acumular más material orgánico que se desprende gradualmente de los dientes y se deposita en estas áreas.

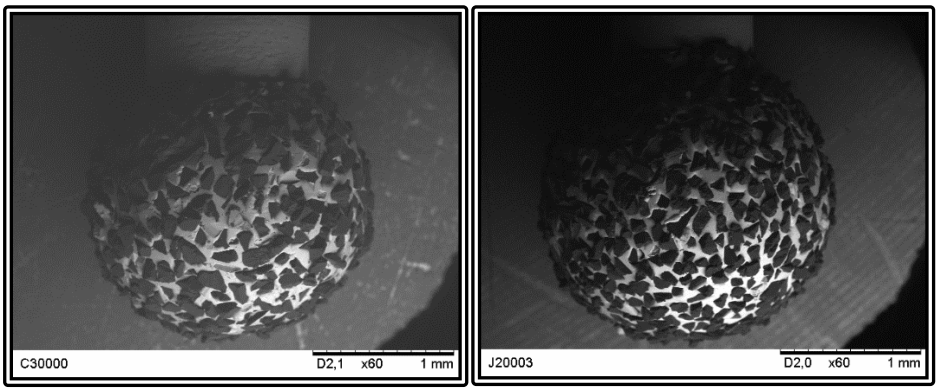


Figura 24. Micrografías SEM de la broca (a) C3 y (b) J3 a 0min de uso, 60x.

En general, se observa que los diámetros medidos en ambos ejes se redujeron en su longitud, de acuerdo al comportamiento esperado debido al desgaste. Sin embargo, la acumulación de material orgánico en los espacios entre los cristales de diamante provoca un aumento de la masa de la broca. Las temperaturas alcanzadas en la interfase debidas a la fricción entre la broca y el diente podrían generar una leve sinterización del cerámico, lo que aumentaría la dureza y las propiedades mecánicas de la capa depositada, provocando que el material base de la broca y los cristales de diamante se desgasten a una tasa menor. Además, esta capa es muy densa, ya que se llenan todos los espacios vacíos debido a esfuerzos mecánicos de compresión que se generan en la interfase. A pesar de esto, el cambio en las medidas registrado es muy

pequeño y varía de acuerdo a muchos factores que se encuentran fuera del alcance de este estudio (velocidad de revolución, propiedades mecánicas de cada pieza extraída, tiempo y temperatura de esterilización de las brocas, presión del chorro de agua para remover depósitos, entre otros), por lo que no se pueden tomar estas variables como los parámetros principales para identificar mecanismos de desgaste.

Por otra parte, el desgaste que se aprecia no es proporcional al tiempo de uso. Como se mencionó anteriormente, durante los primeros 30min de uso, las brocas presentaron un desgaste excesivo en comparación con los últimos 15min de uso, en los cuales la tasa de desgaste no fue tan acelerada. Puede advertirse en la figura 25 que la acumulación de material orgánico no presenta un incremento apreciable de los 30 a los 45min, como sí se observa de los 0 a los 30min de uso. Este comportamiento se explica debido a que, inicialmente, se debe desgastar el esmalte, que es el tejido más duro del diente y se continúa, luego a la dentina y la pulpa, que poseen durezas considerablemente más bajas. Como se describirá más adelante, esta acumulación de material incide en el desgaste de las brocas al no tener las mismas propiedades abrasivas que los cristales de diamante. Los resultados presentados en la tabla 6 muestran las medidas de microdureza Vickers de la dentina y el esmalte de las piezas extraídas que se utilizaron en este estudio, según los procedimientos especificados en las normas ASTM C1327-15 y E384-16 (Anexos I y II). Estos valores coinciden con los reportados en investigaciones previas. [4] [3] [12]

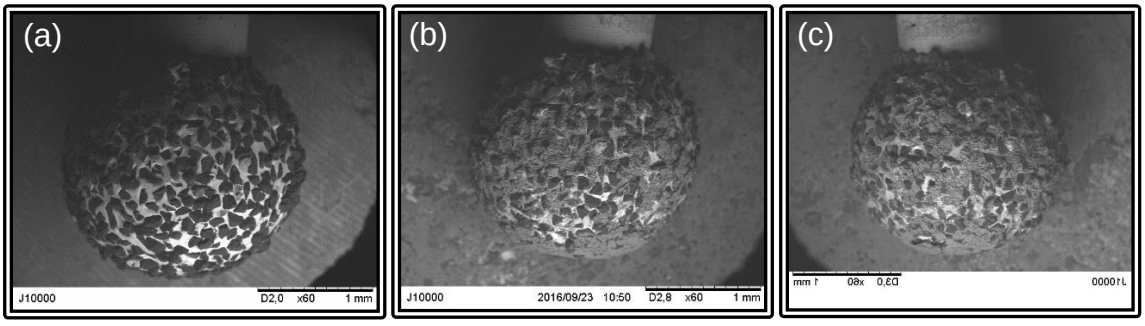


Figura 25. Micrografías SEM de la broca J1 (a) a 0min, (b) 30min y (c) 45min de uso, 60x.

Tabla 6.Dureza de la dentina y el esmalte medidas en una pieza molar extraída.

Tejido	Medición	HV	MPA	GPA	Promedio
Dentina	1	41,0	402,1	-	500,0 MPa
	2	63,3	620,8	-	
	3	55,9	548,2	-	
	4	43,6	427,6	-	
Esmalte	1	271,9	-	2,7	2,8 GPa
	2	419,7	-	4,1	
	3	262,8	-	2,6	
	4	166,6	-	1,6	

Regev, Judes y Ben-Hanan (2010), proponen e identifican 4 mecanismos en los elementos activos que inciden en su desgaste, su deterioro gradual y su eficiencia de corte, siento estos fenómenos parámetros fácilmente observables para advertir el desgaste.

En primer lugar, mencionan el desgaste en los cristales de diamante debido a la abrasión. El proceso inicia con el despuntado de los vértices del cristal y continúa con una disminución en su volumen y masa. [11] Este mecanismo se observó en el presente estudio, como se muestra en la figura 26. Para observar el deterioro de un cristal determinado, se utilizó una broca aparte en la preparación de una cavidad dental, la cual fue fotografiada antes y después de su uso en el mismo punto de referencia. Se puede percibir que el cristal en cuestión (círculo blanco) redujo sus dimensiones después de ser utilizado. El grado de desgaste varía de un cristal a otro dependiendo de su orientación y volumen.

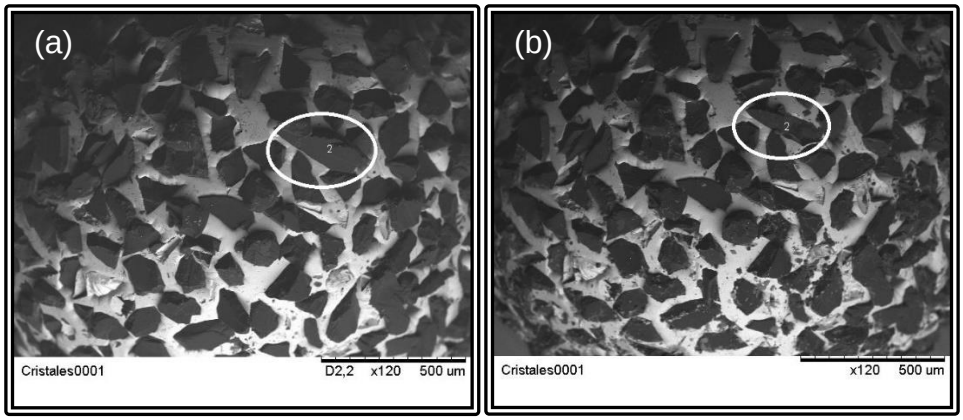


Figura 26. Cambio en el tamaño de un cristal de diamante (a) antes y (b) después de uso, 120x.

El segundo mecanismo observado es el desprendimiento total de cristales de diamante de la matriz, como se muestra en la figura 27, la cual evidencia el arranque de las partículas de cristal de la matriz. Se advierte con claridad la huella dejada por un cristal que fue desprendido durante la operación de la broca. Esta huella no es muy profunda, lo que significa que el cristal no estaba lo suficientemente embebido en la matriz como para mantenerse unido a ella en condiciones de operación. Esto refleja lo expuesto por Siegel y von Franhoufer (1999), quienes expusieron que, de existir insuficiente deposición de matriz de níquel, la tendencia al desprendimiento de las partículas de diamante aumentaba. Idealmente, la matriz de metal electrodepositado debería cubrir un 50-60% del fragmento de diamante.

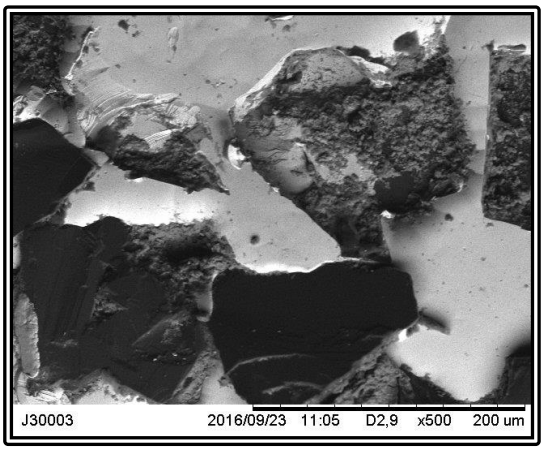


Figura 27. Micrografía SEM de la broca J3 a 30min de uso, 500x.

Otro fenómeno observado es la obstrucción de los espacios entre cristales por los residuos de trituración del tejido dental. En la figura 28a se observa la broca C1 antes de su primer uso, donde la matriz se denota en un tono claro; después de 30min de uso (figura 28b), se puede detectar la presencia de un material diferente a la matriz, el cual se observa en un tono más oscuro. Este material corresponde a las partículas desprendidas de las piezas dentales que no fueron arrastradas por el flujo de agua a presión. Con cada uso, más material cerámico es acumulado hasta casi cubrir los diamantes, como se nota en la figura 29. En adelante, la abrasión generada en las piezas dentales va a ser ocasionada por esta capa acumulada y no por los cristales, lo que resultará en cavidades muy rugosas y de baja calidad superficial.

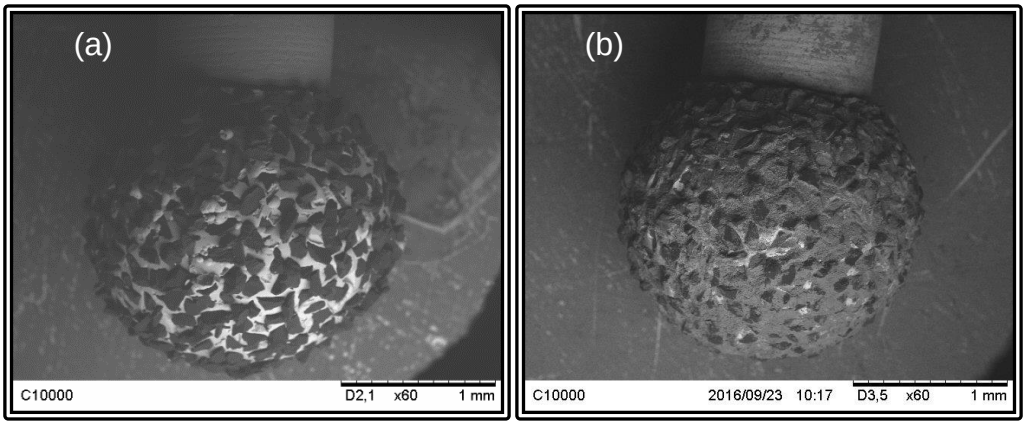


Figura 28. Micrografías SEM de la broca C1 (a) a 0min y (b) a 30min de uso, 60x.

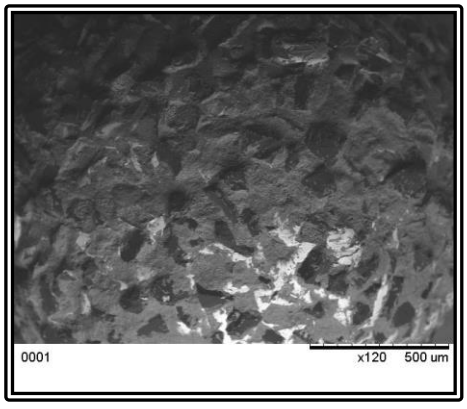


Figura 29. Micrografía SEM de la broca J2 a 45min de uso,120x.

Finalmente, el cuarto mecanismo descrito por Regev, Judes y Ben-Hanan (2010) es la degradación de la matriz de níquel. Este fenómeno es el que menos contribuye al total del desgaste en la broca. La figura 29, muestra el agrietamiento generado por la remoción de una partícula de diamante. Las grietas se extienden desde los vértices de la huella en el material de la matriz. Otras formas de degradación de la matriz que no fueron observadas en este trabajo, son la corrosión causada por las sustancias esterilizadoras o el desgaste abrasivo, que se presenta una vez que los diamantes han sido reducidos exhaustivamente.

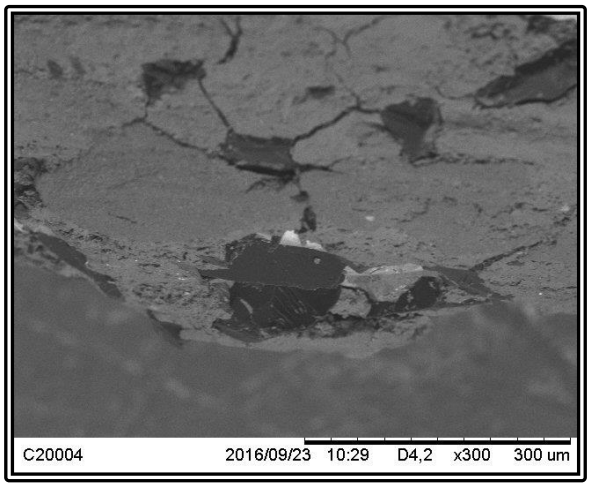


Figura 30. Micrografía SEM de la broca C2 a 30min de uso,300x.

4.1 Cálculo de costos

La clínica realiza pedidos mensuales de 10 brocas. De acuerdo a la cotización realizada por el Depósito Dental Izquierdo, el cual distribuye únicamente la marca Coltène, una broca de las características empleadas en este estudio cuesta ¢1 808. La marca Jota es distribuida en el país por la empresa Prisma Dental Supply, la cual cotizó las brocas de la misma clase en ¢1 636. La inversión mensual de los pedidos sería de ¢18 080 para 10 brocas Coltène, o ¢16 360 para las brocas marca Jota.

5. Conclusiones

Como conclusiones del estudio realizado, pueden citarse las siguientes:

1) El cambio en la masa y en la medida de los diámetros de las brocas no representaron variables significativas en la cuantificación del desgaste.
2) Se identificó el material de la matriz metálica y se estableció una relación entre su composición química y las diversas propiedades requeridas por las brocas dentales para su correcta operación.
3) Se observaron los mismos mecanismos de desgaste en las brocas de ambas marcas (Jota y Coltène), las cuales exhibieron una tasa de desgaste muy similar.
4) Es necesario un estudio más extenso, que integre todas las variables presentes durante la operación de una broca dental (como edad del paciente, hábitos alimenticios y sociales, propiedades mecánicas del diente, velocidad de la broca, presión del agua de arrastre, etc.), para determinar con mayor precisión el efecto de los mecanismos de desgaste.
5) En términos económicos, las brocas marca Jota presentan una mayor rentabilidad ya que su precio unitario es menor que el de las brocas marca Coltène.

6. Recomendaciones

Algunas medidas pueden ser implementadas para mejorar el rendimiento de las brocas y la calidad de los servicios brindados por la clínica; estas son:

1) Utilizar continuamente el flujo de agua a una mayor presión, que arrastre la mayoría de partículas desprendidas del diente, para evitar la aglomeración entre los diamantes de las brocas.
2) Utilizar las brocas dentro del rango de revoluciones establecido por el fabricante (impreso en los empaques), para garantizar una vida útil prolongada.
3) Evitar la esterilización con sustancias ácidas para prevenir la corrosión acelerada de la matriz y el subsecuente deterioro de la broca.
4) Utilizar brocas marca Jota, ya que presentan un deterioro similar y un costo más bajo respecto a las brocas marca Coltène.

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Designation: C1327 – 15

Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics[1]

This standard is issued under the fixed designation C1327; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the Vickers indentation hardness of advanced ceramics. In this test, a pointed, square base, pyramidal diamond indenter of prescribed shape is pressed into the surface of a ceramic with a predetermined force to produce a relatively small, permanent indentation. The surface projection of the two diagonals of the permanent indentation is measured using a light microscope. The average diagonal size and the applied force are used to calculate the Vickers hardness, which represents the material's resistance to penetration by the Vickers indenter. Hardness is computed as the ratio of the force to the contact surface area.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *Units*—When Knoop and Vickers hardness tests were developed, the force levels were specified in units of grams-force (gf) and kilograms-force (kgf). This standard specifies the units of force and length in the International System of Units (SI); that is, force in newtons (N) and length in mm or μm. However, because of the historical precedent and continued common usage, force values in gf and kgf units are occasionally provided for information. This test method specifies that Vickers hardness be reported either in units of GPa, or a dimensionless Vickers hardness number that has implied units of kgf/mm^2.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E4 Practices for Force Verification of Testing Machines

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E384 Test Method for Knoop and Vickers Hardness of Materials

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

IEEE/ASTM SI 10 Standard for the Use of the International System of Units (SI): The Modern Metric System.

2.2 *European Standard:*

CEN ENV 843-4 Advanced Technical Ceramics, Monolithic Ceramics, Mechanical Properties at Room Temperature, Part 4: Vickers, Knoop and Rockwell Superficial Hardness[3]

2.3 *Japanese Standard:*

JIS R 1610 Testing Method for Vickers Hardness of High Performance Ceramics[4]

2.4 *ISO Standard:*

ISO 6507/2 Metallic Materials—Hardness test—Vickers test—Part 2: HV0.2 to less than HV5[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *Vickers hardness number (HV), n*—an expression of hardness obtained by dividing the force applied to a Vickers indenter by the surface area of the permanent impression made by the indenter.

[1] This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Mechanical Properties and Performance.

Current edition approved Jan. 1, 2015. Published March 2015. Originally approved in 1996. Last previous edition approved in 2008 as C1327–08. DOI: 10.1520/C1327-15.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from European Committee for Standardization (CEN), 36 rue de Stassart, B-1050, Brussels, Belgium, http://www.cenorm.be.

[4] Available from Japanese Standards Organization (JSA), 4-1-24 Akasaka Minato-Ku, Tokyo, 107-8440, Japan, http://www.jsa.or.jp.

[5] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

3.1.2 *Vickers indenter, n*—a square-based pyramidal-shaped diamond indenter with face angles of 136° 00′.

4. Summary of Test Method

4.1 This test method describes an indentation hardness test using a calibrated machine to force a pointed, square base, pyramidal diamond indenter having specified face angles, under a predetermined force, into the surface of the material under test and to measure the surface-projected diagonals of the resulting impression after removal of the force.

NOTE 1—A general description of the Vickers indentation hardness test is given in Test Method E384. The present method is very similar, has most of the same requirements, and differs only in areas required by the special nature of advanced ceramics. This test method also has many elements in common with standards ENV 843-4 and JIS R 1610, which are also for advanced ceramics.

5. Significance and Use

5.1 For advanced ceramics, Vickers indenters are used to create indentations whose surface-projected diagonals are measured with optical microscopes. The Vickers indenter creates a square impression from which two surface-projected diagonal lengths are measured. Vickers hardness is calculated from the ratio of the applied force to the area of contact of the four faces of the undeformed indenter. (In contrast, Knoop indenters are also used to measure hardness, but Knoop hardness is calculated from the ratio of the applied force to the projected area on the specimen surface.)

5.2 Vickers indentation hardness is one of many properties that is used to characterize advanced ceramics. Attempts have been made to relate Vickers indentation hardness to other hardness scales, but no generally accepted methods are available. Such conversions are limited in scope and should be used with caution, except for special cases where a reliable basis for the conversion has been obtained by comparison tests.

5.3 Vickers indentation diagonal lengths are approximately 2.8 times shorter than the long diagonal of Knoop indentations, and the indentation depth is approximately 1.5 times deeper than Knoop indentations made at the same force.

5.4 Vickers indentations are influenced less by specimen surface flatness, parallelism, and surface finish than Knoop indentations, but these parameters must be considered nonetheless.

5.5 Vickers indentations are much more likely to cause cracks in advanced ceramics than Knoop indentations. The cracks may influence the measured hardness by fundamentally altering the deformation processes that contribute to the formation of an impression, and they may impair or preclude measurement of the diagonal lengths due to excessive damage at the indentation tips or sides.

5.6 A full hardness characterization includes measurements over a broad range of indentation forces. Vickers hardness of ceramics usually decreases with increasing indentation size or indentation force, as shown in Fig. 1. The trend is known as the indentation size effect (ISE). Hardness approaches a plateau constant hardness at sufficiently large indentation size or forces. The test forces or loads that are needed to achieve a constant hardness vary with the ceramic. The test force specified in this standard is intended to be sufficiently large that hardness is either close to or on the plateau, but not so large as to introduce excessive cracking. A comprehensive characterization of the ISE is recommended but is beyond the scope of this test method, which measures hardness at a single, designated force.

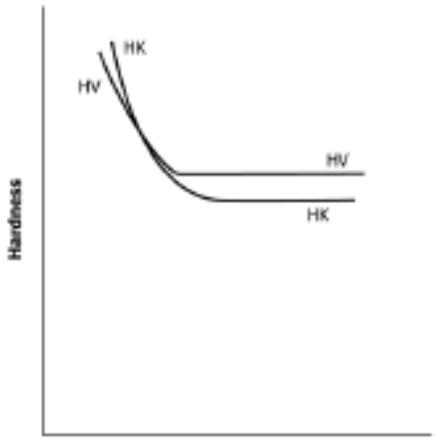


FIG. 1 Indentation Size Effect (ISE) Curves for a Ceramic (Sometimes they continuously approach a plateau hardness at larger forces, but sometimes they can have a shift or step if cracking occurs.)

6. Interferences

6.1 Cracking from the indentation tips can interfere with determination of tip location and thus the diagonal length measurements.

6.2 Cracking or spalling around the Vickers impression may occur and alter the shape and clarity of the indentation, especially for coarse-grained ceramics whereby grains may cleave and dislodge. The cracking may occur in a time-dependent manner (minutes or hours) after the impression is made.

6.3 Porosity (either on or just below the surface) may interfere with measuring Vickers hardness, especially if the indentation falls directly onto a large pore or if the indentation tip falls in a pore.

6.4 At higher magnifications in the optical microscope, it may be difficult to obtain a sharp contrast between the indentation tip and the polished surface of some advanced ceramics. This may be overcome by careful adjustment of the lighting as discussed in Test Method E384.

7. Apparatus

7.1 *Testing Machines:*

7.1.1 There are three general types of machines available for conducting this test. One type is a self-contained unit built for this purpose that uses deadweights (masses) on a pan or lever

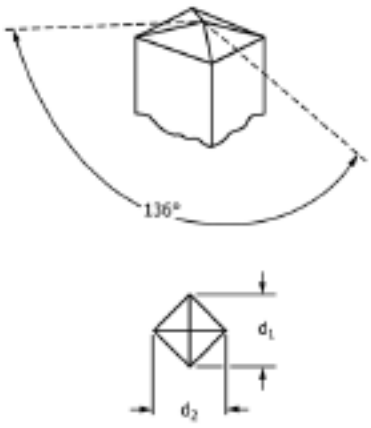


FIG. 2 Vickers Indenter

beam to carefully apply force to the test piece. There is no load cell to record the force during the test sequence. The machine has a built-in compound optical microscope for measuring the indentation sizes. The second type is an accessory to existing compound optical microscopes. Usually, this second type is fitted on an inverted-stage microscope. The third, more modern type, is a self-contained unit built for this purpose which has a built-in load cell that controls a ram or crosshead that moves the indenter into contact with the test piece. The peak force and rate of force application can be controlled by a closed-loop feedback circuit. The machine has a built-in compound optical microscope for measuring the indentation sizes. Descriptions of the various machines are available (**1-3**).[6]

7.1.2 Design of the machine should be such that the force application rate, dwell time, and applied force can be set within the limits set forth in 10.5. It is an advantage to eliminate the human element whenever possible by appropriate machine design. The machine should be designed so that vibrations induced at the beginning of a test will be damped out by the time the indenter touches the sample.

7.1.3 The calibration of the balance beam or force application system should be checked monthly or as needed. Indentations in standard reference materials may also be used to check calibration when needed.

7.2 *Indenter:*

7.2.1 The indenter shall meet the specifications for Vickers indenters. See paragraph A1.3.5.1 of Test Method E384. The four edges formed by the four faces of the indenter shall be sharp. Chamfered edges (as in Ref (**4**)) are not permitted. The tip offset shall be not more than 0.5 µm in length.

7.2.2 Fig. 2 shows the indenter. The depth of the indentation is ⅐ the length of the diagonal. The indenter has an angle between opposite faces of 136° 0 min (±30 min).

7.2.3 The diamond should be examined periodically; and if it is loose in the mounting material, chipped, or cracked, it shall be replaced.

NOTE 2—This requirement is from Test Method E384 and is especially pertinent to Vickers indenters used for advanced ceramics. Vickers indenters are often used at high loads in advanced ceramics in order to create cracks. Such usage can lead to indenter damage. The diamond indenter can be examined with a scanning electron microscope. Indenters may also be inspected with an optical microscope with at least 500× power, but care should be taken to avoid damaging the microscope lens. Indentations can be made into soft copper to help determine if a chip or crack is present. A visual inspection of the resulting indentation may be sufficient to verify the absence of defects from the shape of indentations performed on test blocks.

7.3 *Measuring Microscope:*

7.3.1 The measurement system shall be constructed so that the length of the diagonals can be determined with errors not exceeding ± 0.5 µm (±0.0005 mm).

NOTE 3—Stage micrometers with uncertainties less than this shall be used to establish calibration constants for the microscope. See Test Method E384, paragraph A1.3.3, Verification of the Indentation Measuring System. Ordinary stage micrometers that are used for determining the approximate magnification of photographs may be too coarsely ruled or may not have the required accuracy and precision.

7.3.2 The numerical aperture (NA) of the objective lens shall be between 0.60 and 0.90.

NOTE 4—The apparent length of a Vickers indentation increases as the resolving power and NA of a lens increases. However, the variation is much less than that observed in Knoop indentations (**2**), (**5**), (**6**). The range of NA specified by this test method corresponds to 40 to 100× objective lenses. The higher power lenses may have higher resolution, but the contrast between the indentation tips and the polished surface may be less. This numerical aperture requirement is similar to, but more specific than that in Test Method E384. The requirement is different because many white or grey ceramics are transparent or translucent, and tip imaging is more difficult.

7.3.3 A filter may be used to provide monochromatic illumination. Green filters have proved to be useful.

7.3.4 If indentation diagonal sizes are measured from digital images acquired from a digital camera, then follow the manufacturer's guidelines for use of the camera, the computer monitor, and the software. It is strongly recommended to use a calibrated stage micrometer to verify the precision and accuracy of the length measuring procedure. The camera pixel count, the monitor pixel count and resolution, and the length measuring software shall be such that the requirements of 7.3.1 can be met.

8. Test Specimens

8.1 The Vickers indentation hardness test is adaptable to a wide variety of advanced ceramic specimens. In general, the accuracy of the test will depend on the smoothness of the surface and, whenever possible, ground and polished specimens should be used. The back of the specimen shall be fixed so that the specimen cannot rock or shift during the test.

8.1.1 *Thickness*—As long as the specimen is over ten times as thick as the indentation depth, the test will not be affected. In general, if specimens are at least 0.50 mm thick, the hardness will not be affected by variations in the thickness.

8.1.2 *Surface Finish*—Specimens should have a ground and polished surface. The roughness should be less than 0.1 µm

[6] The boldface numbers in parentheses refer to the list of references at the end of this test method.

rms. However, if one is investigating a surface coating or treatment, one cannot grind and polish the specimen.

NOTE 5—This requirement is necessary to ensure that the surface is flat and that the indentation is sharp. Residual stresses from polishing are of less concern for most advanced ceramics than for glasses or metals. References (7) and (8) report that surfaces prepared with 1 µm or finer diamond abrasive had no effect on measured ceramic hardness. Hardness was only affected when the surface finish had an optically resolvable amount of abrasive damage (7). (Extra caution may be appropriate during polishing of transformation toughening ceramics, such as some zirconias, since the effect upon hardness is not known.)

9. Preparation of Apparatus

9.1 *Verification of Force*—Most of the machines available for Vickers hardness testing use a loaded beam. This beam shall be tested for zero force. An indentation should not be visible with zero force, but the indenter should contact the sample. Methods of verifying the force application are given in Practices E4.

9.2 *Separate Verification of Force, Indenter, and Measuring Microscope*—Procedures in Test Method E384, Annex A1, Verification of Knoop and Vickers Hardness Testing Machines and Indenters, may be followed.

9.3 *Verification by Standard Reference Materials*—Standard reference blocks, SRM No. 2831, of tungsten carbide that are available from the National Institute of Standards and Technology[7] can be used to verify that an apparatus produces a Vickers hardness within ±5 % of the certified value.

10. Procedure

10.1 *Specimen Placement*—Place the specimen on the stage of the machine so that the specimen will not rock or shift during the measurement. The specimen surface shall be clean and free of any grease or film.

10.2 *Specimen Leveling:*

10.2.1 The surface of the specimen being tested shall lie in a plane normal to the axis of the indenter. The angle of the indenter and specimen surface should be within 2° of perpendicular.

NOTE 6—Greater amounts of tilting produce nonuniform indentations and invalid test results. 2° tilt will cause an asymmetrical indentation that is just noticeable, and will cause a 1 % error in hardness (9).

10.2.2 If one leg of a diagonal is noticeably longer than the other leg of the same diagonal, resulting in a deformed indentation, misalignment is probably present and should be corrected before proceeding with any measurements. See Test Method E384.

10.2.3 Leveling the specimen is facilitated if one has a leveling device.[8]

10.2.4 If the diagonal legs are unequal as described in 10.2.2 then rotate the specimen 90° and make another indentation in an untested region. If the nonsymmetrical aspect of the indentations has rotated 90°, then the specimen surface is not perpendicular to the indenter axis. If the nonsymmetrical nature of the indentation remains in the same orientation, check the indenter for misalignment or damage.

10.3 *Magnitude of Test Force*—A test force of 9.81 N (1 kgf) is specified. If other forces are used because of a special requirement, or due to cracking problems at 9.81 N, then the reporting procedure of 12.6 shall be used. If additional forces are used (for example to measure the indentation size effect trend), then the reporting procedure of 12.6 shall be used for each data set.

NOTE 7—"Load" and "Force" are used interchangeably in this standard.

10.4 *Clean the Indenter*—The indenter shall be cleaned prior to and during a test series. A cotton swab with ethanol, methanol, or isopropanol may be used. Indenting into soft copper also may help remove debris. After each change, or removal and replacement, of the indenter it is recommended that a verification be performed with a reference test block. At least two preliminary indentations should be made to ensure that the indenter is seated properly. The results of the preliminary indentations shall be disregarded.

NOTE 8—Ceramic powders or fragments from the ceramic test piece can adhere to the diamond indenter.

10.5 *Application of Test Force:*

10.5.1 Start the machine smoothly. The rate of indenter motion prior to contact with the specimen shall be 0.015 to 0.070 mm/s. If the machine is loaded by an electrical system or a dash-pot lever system, it should be mounted on shock absorbers that damp out all vibrations by the time the indenter touches the specimen.

NOTE 9—This rate of loading is consistent with Test Method E384.

10.5.2 The time of application of the full test force shall be 10 to 15 s unless otherwise specified. After the indenter has been in contact with the specimen from this required dwell time, raise it carefully off the specimen to avoid a vibration impact.

10.5.3 The operator shall not bump or inadvertently contact the test machine or associated support (for example, the table) during the period of indenter contact with the specimen.

10.6 *Spacing of Indentations*—Allow a distance of at least four diagonal lengths between the centers of the indentations as illustrated in Fig. 3. If there is cracking from the indentations, the spacing shall be increased to at least five times the length of the cracks, as shown in Fig. 3.

10.7 *Acceptability of Indentations:*

10.7.1 If there is excessive cracking from the indentation tips and sides, or the indentation is asymmetric, the indent shall be rejected for measurement. Fig. 4 provides guidance in this assessment. If the difference of the two diagonal lengths d1 and d2 is more than 5% of the mean value, the result shall be rejected and a check made of the parallelism and flatness of the test piece, and of the alignment of the indenter. If cracking

[7] Available from National Institute of Standards and Technology (NIST), Standard Reference Materials Program, 100 Bureau Dr., Stop 2300, Gaithersburg, MD 20899-2300, http://www.nist.gov.

[8] The sole source of supply of the apparatus known to the committee at this time is the Tukon Tester leveling device, available from the Wilson Division of Instron Corp. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

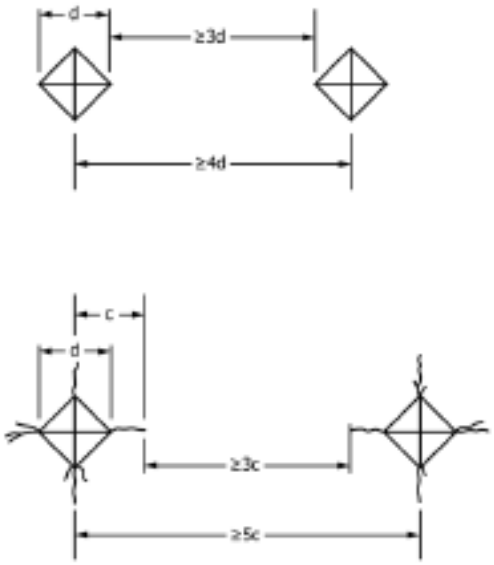


FIG. 3 Closest Permitted Spacing for Vickers Indentations

occurs on most indentations, a lower indentation force (recommended 4.90 N) may be tried.

NOTE 10—If the indentations are still not acceptable, this test method shall not be used to measure hardness. It is recommended that hardness be evaluated by the Knoop hardness method.

10.7.2 If an indentation tip falls in a pore, the indentation shall be rejected. If the indentation lies in or on a large pore, the indent shall be rejected.

NOTE 11—In many ceramics, porosity may be small and finely distributed. The indentations will intersect some porosity. The measured hardness in such instances properly reflects a diminished hardness relative to the fully dense advanced ceramic. The intent of the restrictions in 10.6 is to rule out obviously unsatisfactory or atypical indentations for measurement purposes.

10.7.3 If the impression has an irregularity that indicates the indenter is chipped or cracked, the indent shall be rejected and the indenter shall be replaced.

10.8 In some materials, cracking around the indent may occur in a time dependent manner. If this occurs, the indentation size measurements specified in Section 11 should be made as soon as is practical after the indentation is made. That is, each indent should be measured immediately after it is made (instead of making five or ten indentations and then measuring them).

10.9 *Location of Indentations*—Indentations shall be made in representative areas of the advanced ceramic microstructure. They shall not be restricted to high density regions if such regions exist.

10.10 *Number of Indentations*—For homogeneous and fully dense advanced ceramics, at least five and preferably ten acceptable indentations shall be made. If the ceramic is multiphase, not homogeneous, or not fully dense, ten acceptable indentations shall be made.

11. Measurement of Indentation Size

11.1 The accuracy of the test method depends to a very large extent on this measurement, since hardness depends upon the inverse square of the diagonal size.

11.1.1 If the measuring system contains a light source, take care to use the system only after it has reached equilibrium temperature. This is because the magnification of a microscope depends on the tube length.

11.1.2 Calibrate the measuring system carefully with an accurate and precision stage micrometer or with an optical grating.

11.1.3 Adjust the illumination and focusing conditions carefully as specified in Test Method E384 to obtain the optimum view and clarity of the impression. Proper focus and illumination are critical for accurate and precise readings. Both indentation tips shall be in focus at the same time. Do not change the focus once the measurement of the diagonal length has begun.

NOTE 12—The lighting intensity and the settings of the field and aperture diaphragms can have a noticeable effect upon the apparent location of the tips in Vickers indentations. Consult the manufacturer's guidelines for optimum procedures. Additional information is presented in Test Method E384. In general, the field diaphragm can be closed so that it barely enters or just disappears from the field of view. The aperture diaphragm can be closed in order to reduce glare and sharpen the image, but it should not be closed so much as to cause diffraction that distorts the edges of the indentation. These requirements are especially important with white or grey ceramics that may be transparent or translucent, and tip imaging may be more difficult.

NOTE 13—Uplift and curvature of the sides of the impressions may be substantial in impressions in advanced ceramics, which may cause the sides of the impression to be slightly out of focus. The tips of the impression shall be focused on for measurement of the indentation diagonals. It may be helpful to focus on a small microstructural feature on the flat specimen surface just beyond the indentation tips.

11.1.4 If either a measuring microscope or a filar micrometer eyepiece is used, always rotate the drum in the same direction to eliminate backlash errors.

11.1.5 Follow the manufacturer's guidelines for the use of crosshairs or graduated lines. To eliminate the influence of the thickness of the line, always use the same edge of the crosshair or graduation line. **Caution**—Serious systematic errors can occur due to improper crosshair usage. Procedures vary considerably between different equipment. In nearly all instances, the crosshairs should not be placed entirely over or fully cover the indentation tip as shown in part (a) of Fig. 5. The indentation tip should be just visible in the fringe of light on the side of the crosshair or graduated line as shown in part (b) of Fig. 5 or part (c) of Fig. 5. In some measuring systems with twin crosshairs, the measurement is made with the inside edge of the two lines as shown in part (b) of Fig. 5. In other measuring systems, particularly those with a single moveable crosshair, the measurement is made with the same side of the crosshair as shown in part (c) of Fig. 5.

11.1.6 Read the two diagonals of the indent to within 0.25 μm (0.00025) mm and determine the average of the diagonal lengths.

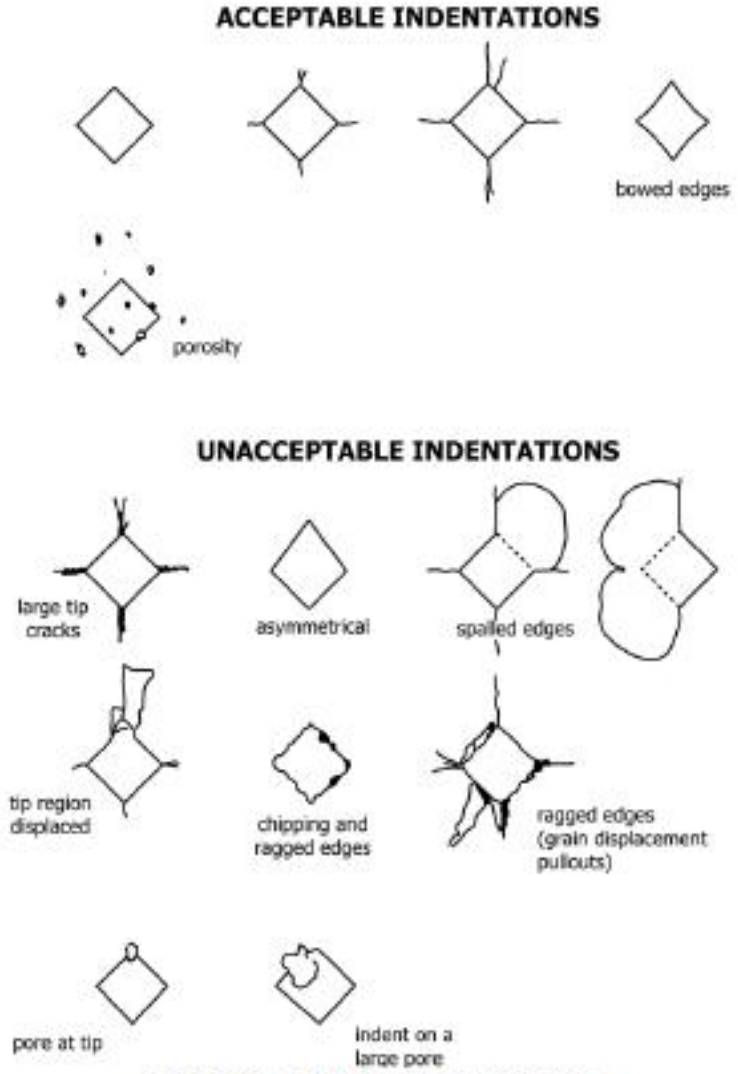


FIG. 4 Guidelines for the Acceptance of Indentations

11.1.7 Use the same filters in the light system at all times. Usually a green filter is used.

11.1.8 For transparent or translucent ceramics, where contrast is poor, the specimen may be coated (for example, a gold/palladium coating) to improve the measurability of the indents (**4**). Such coatings shall be less than 50 nm thick and shall be applied after the indentations have been made. Never indent into coatings made to enhance visibility.

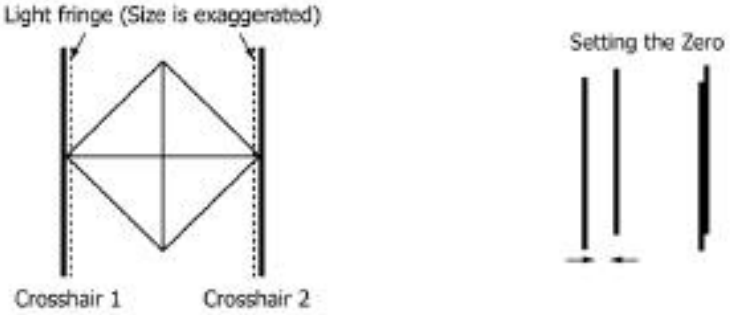
12. Calculation

12.1 Vickers hardness may be calculated and reported either in units of GPa (12.2) or as a dimensionless Vickers hardness number (12.3).

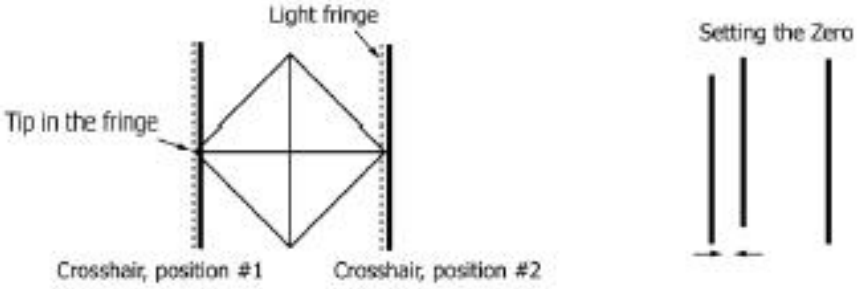
12.2 The Vickers hardness with units of GPa is computed as follows:

$$HV = 0.0018544\,(P/d^2) \quad (1)$$

(a) INCORRECT. Crosshair completely covers the tip.



(b) CORRECT. Double crosshair measurement system, whereby the indentation is intended to be measured between two crosshairs or measuring lines. Indentation tips should be on the inside edge (in the fringe) of each crosshair. The measuring system is zeroed by bringing the inside measuring line inside edges together as shown on the right.



(c) CORRECT. Single crosshair and some double crosshair measurement systems. The indentation tip is on the same side of the crosshair line(s). The measuring system is zeroed with the tip on the same side of one line for a single crosshair system, or with both lines superimposed in a double crosshair system as shown on the right.

FIG. 5 Crosshair Measurement

where:

P = force, N, and
d = average length of the two diagonals of the indentation, mm.

NOTE 14—This computation and set of units are in accordance with the recommendations of IEEE/ASTM SI 10.

12.3 The Vickers hardness number is computed as follows:

$$HV = 1.8544\,(P/d^2) \tag{2}$$

where:

P = force, kgf, and
d = average length of the two diagonals of the indentation, mm.

NOTE 15—This computation is consistent with Test Method E384.

Alternately, the Vickers hardness number also may be computed as follows:

$$HV = (0.102)(1.8544)(P/d^2) \tag{3}$$

where:

P = force, N, and
d = average length of the two diagonals of the indentation, mm.

NOTE 16—This computation is consistent with ISO 6507/2, ENV 843-4, and JIS R 1610.

NOTE 17—Eq 2 and Eq 3 compute the Vickers hardness number, which is a dimensionless number; for example, $HV = 1500$. HV formerly had been assigned units of kgf/mm^2. Eq 2 and Eq 3 produce the same Vickers hardness number.

NOTE 18—The factor 0.102 in Eq 3 becomes necessary through the introduction of the SI unit newton for the test force instead of kilogram-force to avoid changing the value of the Vickers hardness number from its traditional units.

12.4 The mean hardness, $\overline{HV}$, is:

$$\overline{HV} = \frac{\sum HV_n}{n} \tag{4}$$

where:

HV_n = HV obtained from nth indentation and
n = number of indentations.

12.5 The standard deviation, S, is:

$$S = \sqrt{\frac{\sum \left(\overline{HV} - HV_n\right)^2}{n-1}} \tag{5}$$

12.6 The hardness symbol HV shall be supplemented by a number indicating the test force used, expressed in newtons multiplied by 0.102 (and therefore equal to the test force expressed in kilograms-force), and optionally a number indicating the duration of test force applications in seconds. So, for example, $HV1/15$ means the Vickers hardness for an applied test force of 9.81 N (1 kgf) applied for 15 s at full load.

13. Report

13.1 The report shall include the following information:

13.1.1 Mean HV,
13.1.2 Test load,
13.1.3 Duration of test load,
13.1.4 Standard deviation,
13.1.5 Test temperature and humidity,
13.1.6 Number of satisfactory indentations measured, as well as the total number of indents made,
13.1.7 Surface conditions and surface preparation,
13.1.8 Thermal history of the sample,
13.1.9 The extent of cracking (if any) observed, and
13.1.10 Deviations from the specified procedures, if any.
13.1.11 Data on the indentation size effect trend if hardness is measured over a range of indentation forces.

14. Precision and Bias

14.1 The precision and bias of microhardness measurements depend on strict adherence to the stated test procedure and are influenced by instrumental and material factors and indentation measurement errors.

14.2 The consistency of agreement for repeated tests on the same material is dependent on the homogeneity of the material, repeatability and reproducibility of the hardness tester, and consistent, careful measurements of the indents by a competent operator.

14.3 Instrumental factors that can affect test results include accuracy of loading, inertia effects, speed of loading, vibrations, the angle of indentation, lateral movement of the indenter or sample, indentation, and indenter shape deviations. Results are particularly sensitive to vibration or impact, which will produce larger indents and lower apparent hardness results.

14.4 The largest source of error or uncertainty in hardness usually arises from the error and uncertainty in the measurement of the diagonal length.

14.4.1 The harder the material, the smaller the indent size is. Therefore, hardness uncertainties are usually greater for harder materials.

14.4.2 Diagonal length measurement errors include inaccurate calibration of the measuring device, inadequate resolving power of the objective, insufficient magnification, operator bias in sizing the indents, poor image quality, and nonuniform illumination. These can contribute to both bias and precision errors.

14.4.3 The numerical aperture (NA) of the objective lens determines the maximum useful magnification and the resolving power of the microscope. The higher the NA of the lens, the longer the indentation will appear. This limited resolution leads to a bias error since the microscope is not able to resolve the exact tip and thus leads to underestimates of the true length. The theoretical shortening is estimated to be $\lambda/2NA$, where λ is the wavelength of the light used (2), (5). Experimental evidence indicates that actual shortening is less than this, but the use of different NA objective lenses will contribute to a reproducibility (between-laboratory) uncertainty of less than ± 0.2 µm (5), (6). (This error is substantially less for Vickers indentations than for Knoop indentations.)

14.5 A round robin was conducted to evaluate the suitability of tungsten carbide-cobalt specimens as standard hardness test blocks[9] (1, 2). The results of this eleven-laboratory round robin can be used to evaluate the precision of Vickers hardness measurements for a hard material (~15 GPa) that does not

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C28-1004. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Precision of Diagonal Length Measurements Estimated from an Interlaboratory Round Robin Project (10, 11)

Load, P (N)	Number of Laboratories	Grand Average Diagonal Length, d (μm)	Within-Laboratory Repeatability			Between-Laboratory Reproducibility		
			Standard Deviation (μm)	Expanded Uncertainty[A] (μm)	Coefficient of Variation, %	Standard Deviation (μm)	Expanded Uncertainty[A] (μm)	Coefficient of Variation, %
9.81[B]	10	34.5	0.2	0.6	0.6	1.1	2.9	3.0
9.81[C]	8	34.6	0.2	0.6	0.6	1.0	2.7	2.8

[A] Coverage factor of 2.8, corresponding to a 95 % confidence interval.
[B] Indentations made by organizing laboratory. Outlier results from one laboratory deleted.
[C] Indentations made by participating laboratories. Outlier results from two laboratories deleted. One other laboratory did not do this part of the exercise.

pose difficult measuring problems. Within-laboratory repeatability and between-laboratory reproducibility were evaluated in accordance with Practices E177 and E691. The results are listed in Table 1, which shows the repeatability and reproducibility in measured diagonal lengths when using the average diagonal size for each indentation. The hardness repeatability interval when expressed as a percentage is double the diagonal-length repeatability interval. Participants read five indentations made at 9.81 N at the organizing laboratory, and also made and measured five of their own indentations at the same force. They reported the average diagonal size for each of the five indentations and the overall average for all five indentations. Table 1 shows the grand average of all accepted laboratory results. The within-laboratory hardness repeatabilities were 1.2 and 1.3 % (coefficient of variation, COV), respectively. The between-laboratory hardness reproducibilities were 6.1 and 5.6 % (COV), respectively. The reproducibility estimates were made after deleting one or two outlier sets as noted in Table 1. The reproducibility uncertainty includes both the hardness measurement uncertainty and the variations in hardness (±2.8 %, COV) of the eight blocks used in the round robin.

15. Keywords

15.1 advanced ceramics; cracks; indentation; microscope; Vickers hardness

APPENDIX

(Nonmandatory Information)

X1. REVISION HISTORY

X1.1 This standard was originally adopted in February 1996 as C1327-96.

X1.2 In late 1996, revisions were made including a recommendation that gold coatings not be applied before making indentations, new guidance on how to inspect the Vickers indenter for damage, and new guidance on how to use crosshairs on the optical microscope when measuring indentation sizes. The revised standard was designated C1327-96a.

X1.3 In 1999, a revision was made to the definition of "Vickers hardness number." The old definition explicitly stated it was with kilograms force. The new definition was more generic and deleted mention of kilograms force and millimetres. This change matched a similar change in Test Method E384. A definition of a "Vickers indenter" was also added. The revised standard was designated C1327-99.

X1.4 In 2003, four revisions were made including: a change of the lower limit of the compound optical microscope numerical aperture limit from 0.65 to 0.60; addition of information about the indentation size effect; addition of limits of asymmetry of an indentation; and editorial changes such as changing the word "load" to "force." The revised standard was designated C1327-03.

X1.5 In 2008, two new columns of data were added to Table 1 for the interlaboratory round robin project results. The new columns listed the standard deviation values for the repeatability and reproducibility of the diagonal length measurements. The revised standard was designated C1327-08.

X1.6 In 2015, changes were made including: minor adjustments to harmonize this test method with the newly revised Test Method E384; additions to the Scope section to better explain this test method and also the history of the units in use; conversion of a few more "load" to "force" terms; more guidance on inspecting diamond indenters in 7.2.3; more guidance on digital cameras for measuring diagonal sizes in 7.3.4; more guidance on asymmetric indentations in 10.2.4; and a small but important clarification in the fourth sentence of 14.5 that the repeatability and reproducibility uncertainties in Table 1 are for the average diagonal size for a single indentation computed from the two diagonal. One major change is the addition of a new Fig. 1 showing a schematic of the indentation size effect curve. Another major change is an expansion of the types of hardness machines from two to three possible types in 7.1 to include modern machines with load cells and closed loop feedback control of the force application cycle. The revised standard was designated C1327-15.

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Designation: E384 – 16

Standard Test Method for Microindentation Hardness of Materials[1]

This standard is issued under the fixed designation E384; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers determination of the microindentation hardness of materials.

1.2 This test method covers microindentation tests made with Knoop and Vickers indenters under test forces in the range from 9.8×10^{-3} to 9.8 N (1 to 1000 gf).

1.3 This test method includes an analysis of the possible sources of errors that can occur during microindentation testing and how these factors affect the precision, bias, repeatability, and reproducibility of test results.

1.4 Information pertaining to the requirements for direct verification and calibration of the testing machine and the requirements for the manufacture and calibration of Vickers and Knoop reference hardness test blocks are in Test Method E92.

NOTE 1—While Committee E04 is primarily concerned with metals, the test procedures described are applicable to other materials.

1.5 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1326 Test Method for Knoop Indentation Hardness of Advanced Ceramics
C1327 Test Method for Vickers Indentation Hardness of Advanced Ceramics
E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography
E92 Test Method For Vickers Hardness of Metallic Materials
E140 Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness
E175 Terminology of Microscopy
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope
E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques
E2587 Practice for Use of Control Charts in Statistical Process Control

2.2 *ISO Standard:*[3]

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibrating, v*—determining the values of the significant parameters by comparison with values indicated by a reference instrument or by a set of reference standards.

3.2.2 *Knoop hardness number, HK, n*—an expression of hardness obtained by dividing the force applied to the Knoop indenter by the projected area of the permanent impression made by the indenter.

[1] This test method is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.05 on Microindentation Hardness Testing. With this revision the test method was expanded to include the requirements previously defined in E28.92, Standard Test Method for Vickers Hardness Testing of Metallic Material that was under the jurisdiction of E28.06.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.org.

*A Summary of Changes section appears at the end of this standard

3.2.3 *Knoop indenter, n*—a rhombic-based pyramidal-shaped diamond indenter with edge angles of ∠ A = 172° 30′ and ∠ B = 130° 0′ (see Fig. 1).

3.2.4 *microindentation hardness test, n*—a hardness test using a calibrated machine to force a diamond indenter of specific geometry into the surface of the material being evaluated, in which the test forces range from 1 to 1000 gf (9.8 × 10^{-3} to 9.8 N), and the indentation diagonal, or diagonals, are measured with a light microscope after load removal; for any microindentation hardness test, it is assumed that the indentation does not undergo elastic recovery after force removal.

NOTE 2—Use of the term microhardness should be avoided because it implies that the hardness, rather than the force or the indentation size, is very low.

3.2.5 *verifying, v*—checking or testing the instrument to assure conformance with the specification.

3.2.6 *Vickers hardness number, HV, n*—an expression of hardness obtained by dividing the force applied to a Vickers indenter by the surface area of the permanent impression made by the indenter.

3.2.7 *Vickers indenter, n*—a square-based pyramidal-shaped diamond indenter with face angles of 136° (see Fig. 2).

3.3 *Formulae*—The formulae presented in 3.3.1 – 3.3.4 for calculating microindentation hardness are based upon an ideal tester and conditions. The measured value of the microindentation hardness of a material is subjected to several sources of errors. Based on Eq 1-9, variations in the applied force, geometrical variations between diamond indenters, and human errors in measuring indentation lengths will affect the precision of the calculated material hardness. The magnitude of the error that variations of each of these parameters have on the calculated value of a microindentation measurement is discussed in Section 10.

3.3.1 For Knoop hardness tests, in practice, test loads are in grams-force and indentation diagonals are in micrometers. The Knoop hardness number is calculated using the following:

$$HK = 1.000 \times 10^3 \times (P/A_p) = 1.000 \times 10^3 \times P/(c_p \times d^2) \quad (1)$$

or

$$HK = 14229 \times P/d^2 \quad (2)$$

$$c_p = \frac{\tan\frac{\angle B}{2}}{2\tan\frac{\angle A}{2}} \quad (3)$$

where:

P = force, gf,
d = length of long diagonal, μm,
A_p = projected area of indentation, μm ^{2}
$\angle A$ = included longitudinal edge angle, 172° 30′
$\angle B$ = included transverse edge angle, 130° 0′ (see Fig. 1 and,
c_p = indenter constant relating projected area of the indentation to the square of the length of the long diagonal, ideally 0.07028.

3.3.2 The Knoop hardness, kgf/mm^2 is determined as follows:

$$HK = 14.229 \times P_1/d_1^2 \quad (4)$$

where:

P_1 = force, kgf, and
d_1 = length of long diagonal, mm.

3.3.3 The Knoop hardness reported with units of GPa is determined as follows:

$$HK = 0.014229 \times P_1/d_1^2 \quad (5)$$

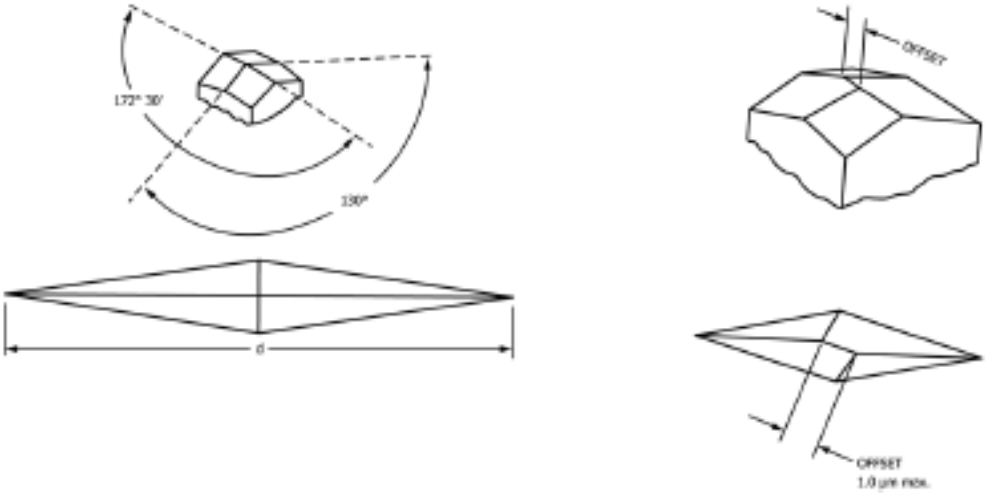


FIG. 1 Knoop Indenter

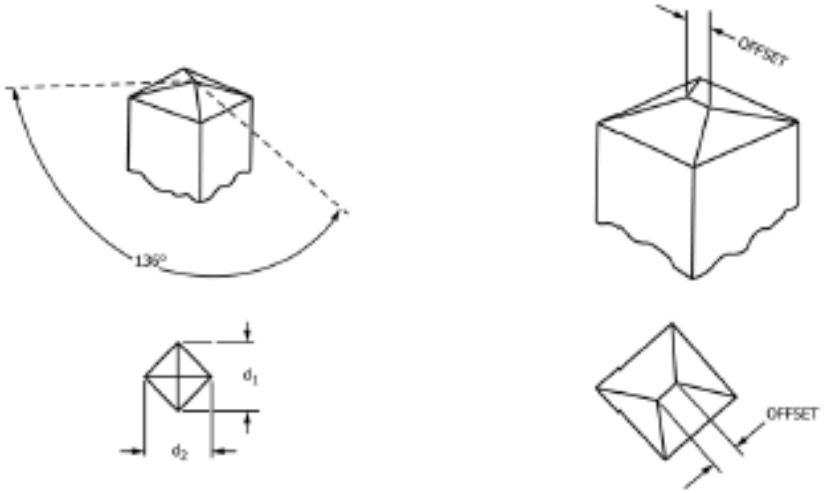


FIG. 2 Vickers Indenter

where:

P_2 = force, N, and
d_2 = length of the long diagonal of the indentation, mm.

3.3.4 For the Vickers hardness test, in practice, test loads are in grams-force and indentation diagonals are in micrometers. The Vickers hardness number is calculated as follows:

$$HV = 1.000 \times 10^3 \times P/A_s = 2.000 \times 10^3 \times P\sin(\alpha/2)/d^2 \quad (6)$$

or

$$HV = 1854.4 \times P/d^2 \quad (7)$$

where:

P = force, gf,
A_s = surface area of the indentation, μm^2,
d = mean diagonal length of the indentation, µm, and
α = face angle of the indenter, 136° 0′ (see Fig. 2).

3.3.5 The Vickers hardness, kgf/mm^2 is determined as follows:

$$HV = 1.8544 \times P_1/d_1^2 \quad (8)$$

where:

P_1 = force, kgf, and
d_1 = mean diagonal length of the indentations, mm.

3.3.6 The Vickers hardness reported with units of GPa is determined as follows:

$$HV = 0.0018544 \times P_2/d_2^2 \quad (9)$$

where:

P_2 = force, N, and
d_2 = mean diagonal length of the indentations, mm.

3.4 Equations for calculating % Error and Repeatability for periodic verification is determined as follows:

$$E = 100\left(\frac{\bar{d} - d_{ref}}{d_{ref}}\right) \quad (10)$$

where:

E = % error in performance of the periodic verification,
$\bar{d}$ = the measured mean diagonal length in µm, and
d_{ref} = the reported certified mean diagonal length, µm.

$$R = 100\left(\frac{d_{max} - d_{min}}{\bar{d}}\right) \quad (11)$$

where:

R = repeatability in performance of the periodic verification,
d_{max} = the longest diagonal length measurement on the standardized test block, µm,
d_{min} = the shortest diagonal length measurement on the standardized test block, µm, and
$\bar{d}$ = the measured mean diagonal length in µm.

4. Summary of Test Method

4.1 In this test method, a hardness number is determined based on the formation of a very small indentation by application of a relatively low force, in comparison to traditional bulk indentation hardness tests.

4.2 A Knoop or Vickers indenter, made from diamond of specific geometry, is pressed into the test specimen surface under an applied force in the range of 1 to 1000 gf using a test machine specifically designed for such work.

4.3 The size of the indentation is measured using a light microscope equipped with a filar type eyepiece, or other type of measuring device (see Terminology E175).

4.4 The Knoop hardness number is based upon the force divided by the projected area of the indentation. The Vickers hardness number is based upon the force divided by the surface area of the indentation.

4.5 It is assumed that elastic recovery does not occur when the indenter is removed after the loading cycle, that is, it is

assumed that the indentation retains the shape of the indenter after the force is removed, but this is not always true. In Knoop testing, it is assumed that the ratio of the long diagonal to the short diagonal of the impression is the same as for the indenter, 7.114, but this is not always true due to elastic recovery.

5. Significance and Use

5.1 Hardness tests have been found to be very useful for materials evaluation, quality control of manufacturing processes and research and development efforts. Hardness, although empirical in nature, can be correlated to tensile strength for many metals and alloys, and is also an indicator of machinability, wear resistance, toughness and ductility.

5.2 Microindentation tests are utilized to evaluate and quantify hardness variations that occur over a small distance. These variations may be intentional, such as produced by localized surface hardening, for example, from shot blasting, cold drawing, flame hardening, induction hardening, etc., or from processes such as carburization, nitriding, carbonitriding, etc.; or, they may be unintentional variations due to problems, such as decarburization, localized softening in service, or from compositional/microstructural segregation problems. Low test forces also extend hardness testing to materials too thin or too small for macroindentation tests. Microindentation tests permit hardness testing of specific phases or constituents and regions or gradients too small for evaluation by macroindentation tests.

5.3 Because microindentation hardness tests will reveal hardness variations that commonly exist within most materials, a single test value may not be representative of the bulk hardness. Vickers tests at 1000 gf can be utilized for determination of the bulk hardness, but, as for any hardness test, it is recommended that a number of indents are made and the average and standard deviation are calculated, as needed or as required.

5.4 Microindentation hardness testing is generally performed to quantify variations in hardness that occur over small distances. To determine these differences requires a very small physical indentation. Testers that create indents at very low test forces must be carefully constructed to accurately apply the test forces exactly at the desired location and must have a high-quality optical system to precisely measure the diagonal (or diagonals) of the small indents. Test forces in the upper range of the force range defined in 1.2 may be used to evaluate bulk hardness. In general, the Vickers indenter is better suited for determining bulk (average) properties as Vickers hardness is not altered by the choice of the test force, from 25 to 1000 gf, because the indent geometry is constant as a function of indent depth. The Knoop indentation, however, is not geometrically identical as a function of depth and there will be variations in Knoop hardness, particularly at test forces <200 gf, over the force range defined in 1.2 (and above this range); consequently, Knoop hardness is not normally used to define bulk hardness, except at 500 gf where E140 gives conversions to other test scales, and Knoop tests should not be performed at test forces above 1000 gf. The majority of Knoop tests of case hardness variations are conducted at forces from 100 to 500 gf. If the test is being conducted to meet a specified bulk hardness value, such as HRC, then most such tests will be conducted with Knoop at a 500 gf load. Because of the large difference between the long and short Knoop diagonals, the Knoop indenter is often better suited for determining variations of hardness over very small distances compared to the Vickers indenter. Vickers and Knoop tests at forces ≤25 gf are susceptible to imprecision due to the difficulty in measuring extremely small indents (<20 μm) by light microscopy with high precision and reproducibility. Tests made at forces ≤25 gf should be considered to be qualitative in nature. Likewise, test forces that create indents <20 μm in length should be avoided whenever possible and should be considered to be qualitative in nature. The success of the specimen preparation procedure in removing preparation-induced damage can, and will, influence test results; this problem becomes more critical as the test force decreases.

6. Apparatus

6.1 *Test Machine*—The test machine must support the test specimen and control the movement of the indenter into the specimen under a preselected test force, and should have a light optical microscope to select the desired test locations and to measure the size of the indentations produced by the test. The plane of the surface of the test specimen must be perpendicular to the axis of the indenter and the direction of the force application. The plane of the test specimen surface must be flat, and free of surface relief, in order to obtain valid, usable test data. The hardness test machine must meet the verification requirements defined in Test Method E92.

6.1.1 *Force Application*—The test machine shall be capable of applying the test forces according to the following:

6.1.1.1 The time from the initial application of the force until the full test force is reached shall not exceed 10 s.

6.1.1.2 The indenter shall contact the specimen at a velocity between 15 and 70 μm/s. Indenter velocity is not usually adjustable by the user.

6.1.1.3 The full test force shall be applied for 10 to 15 s unless otherwise specified.

6.1.1.4 For some applications it may be necessary to apply the test force for longer times. In these instances the tolerance for the time of the applied force is ± 2 s.

6.1.2 *Vibration Control*—During the entire test cycle, the test machine should be protected from shock or vibration. To minimize vibrations, the operator should avoid contacting the machine, or the support table, in any manner during the entire test cycle.

6.2 *Vickers Indenter*—The Vickers indenter normally produces geometrically-similar indentation shapes at all test forces. Except for tests at very low forces that produce indentations with diagonals smaller than about 20 μm, the Vickers hardness number will be the same, within statistical precision limits, as produced using test forces that produce diagonal lengths ≥20 μm, using either a microindentation test machine up to 1000 gf or a macroindentation test machine with test forces ≥ 1 kgf, as long as the material being tested is reasonably homogeneous and the magnification and image quality are optimal (see Appendix X4). For isotropic materials, the two diagonals of a Vickers indentation are equal in size.

Metals/alloys with preferred crystallographic textures may produce distorted indents and invalid or questionable test results. The Vickers indenter must meet the verification requirements defined in Test Method E92.

6.2.1 The ideal Vickers indenter is a highly polished, pointed, square-based pyramidal diamond with face angles of 136° 0′. The effect that geometrical variations of these angles have on the measured values of Vickers hardness is discussed in Section 10.

6.2.2 The four faces of the Vickers indenter shall be equally inclined to the axis of the indenter (within ± 30′) and shall meet at a sharp point. The line of junction between opposite faces (offset) shall be not more than 0.5 µm in length as shown in Fig. 2.

6.3 *Knoop Indenter*—The Knoop indenter does not produce geometrically-similar indentation shapes as a function of test force and indent depth. Consequently, the Knoop hardness will vary with test force (see Appendix X4). Due to its rhombic shape, the indentation depth is shallower for a Knoop indentation compared to a Vickers indentation under identical test conditions. But, for the same test force, the Knoop long diagonal will be substantially longer than the mean of the two Vickers diagonals. The two diagonals of a Knoop indentation are markedly different. Ideally, the long diagonal is 7.114 times longer than the short diagonal, but this ratio is influenced by elastic recovery. Because of its shape, the Knoop indenter is very useful for evaluating hardness gradients or thin coatings. The Knoop test is not recommended for use above a 1 kgf test load. The Knoop indenter must meet the verification requirements defined in Test Method E92.

6.3.1 The Knoop indenter is a highly polished, pointed, rhombic-based, pyramidal diamond (1).[4] The ideal included longitudinal edge angles are 172° 30′ and 130° 0′. The ideal indenter constant, c_p, is 0.07028. The effect that geometrical variations of these angles have on the measured values of Knoop hardness is discussed in Section 10.

6.3.2 The four faces of the Knoop indenter shall be equally inclined to the axis of the indenter (within ± 30′) and shall meet at a sharp point. The line of junction between opposite faces (offset) shall be not more than 1.0 µm in length for indentations greater than 20 µm in length, as shown in Fig. 1. For shorter indentations, the offset should be proportionately less.

6.3.3 Indenters should be examined periodically and replaced if they become worn, dulled, chipped, cracked or separated from the mounting material. Never touch the indenter tip with your finger.

6.4 *Measuring Equipment*—The test machine's measuring device should report the diagonal lengths in 0.1 µm increments for indentations with diagonals from 1 to 200 µm.

NOTE 3—This is the reported length and not the resolution of the system used for performing the measurements. As an example, if a length of 200 µm corresponds to 300 filar units or pixels, the corresponding calibration constant would be 200/300 = 0.66666667. This value would be used to compute diagonal lengths, but the reported length would only be reported to the nearest 0.1 µm.

6.4.1 The optical portion of the measuring device should utilize Köhler illumination. Consult the manufacturer's instruction manual for the adjustments that can be made on your tester.

6.4.2 To obtain maximum resolution, the measuring microscope should have high quality objectives with adequate numerical apertures, a suitable eyepiece, adjustable illumination intensity, adjustable alignment and aperture and field diaphragms. These are adjusted in the same manner as on a reflected light microscope or metallograph. Some systems are now designed using computer monitors and indent length detection by image analysis and may not utilize a traditional eyepiece, but have a projection lens connected to a CCD camera. While a traditional eyepiece has a circular field of view, the computer monitor is rectangular and its height-to-width ratio can vary.

6.4.3 Magnifications should be provided so that the diagonal can be enlarged to greater than 25 % but less than 75 % of the field width. If the computer screen has a 4 to 3 ratio of width to height, or a greater difference between the screen width and height, the maximum field height must be <75% of the width to measure both Vickers diagonals. A 40× or 50× objective may not be adequate for precise measurement of indents <30 µm in length. Measurements of diagonal lengths <20 µm in length with the light microscope may be imprecise, regardless of the objective magnification used, with the problem becoming more acute as the diagonal length decreases below 20 µm.

7. Test Specimen

7.1 For optimum accuracy of measurement, the test should be performed on a flat specimen with a polished surface free of preparation-induced damage. The surface must be free of any problems that could affect the indentation or the subsequent measurement of the diagonals. Conducting tests on non-planar surfaces is not recommended. Results will be affected even in the case of the Knoop test where the radius of curvature is in the direction of the short diagonal.

7.1.1 In all tests, the indentation perimeter, and the indentation tips in particular, must be clearly defined in the microscope field of view.

7.1.2 For best results, the specimen surface should not be etched before making an indentation (2), although etching is often necessary to aid indent location. Deeply etched surfaces will obscure the edge of the indentation, making an accurate measurement of the size of the indentation difficult or impossible. When determining the microindentation hardness of an isolated phase or constituent, or when evaluating segregated compared to non-segregated areas, and other similar situations, a light etch is required to delineate the object or area of interest so that the indentations can be placed in the desired locations. The necessary quality of the required surface preparation does vary with the forces and magnifications used in microindentation hardness testing. The lighter the force and the smaller the indentation size, the more critical is the surface preparation. Some materials are more sensitive to preparation-induced

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

damage than others. In general, face-centered cubic metals (for example, austenitic stainless steels, copper and its alloys, nickel and its alloys, gold and silver) exhibit a larger deformation field around the indent than an indent of the same test force made in a body-centered cubic metal (for example, ferritic and martensitic steels).

7.1.3 Due to the small size of the indentations, special precautions must be taken during specimen preparation. It is well known that improper preparation can alter test results. Specimen preparation must remove any damage introduced during these steps, either due to excessive heating or cold work, for example.

7.1.4 Specimen preparation should be performed in accordance with Guide E3.

7.2 In many instances, it is necessary to mount the specimen for convenience in preparation and for best edge retention. When mounting is required, the specimen must be adequately supported by the mounting medium so that the specimen does not move during force application, such as might happen in an improperly cured polymer mount.

8. Procedure

8.1 Turn on the illumination system and power for the tester.

8.2 Select the desired indenter. If it is necessary to physically change indenters, refer to the manufacturer's instructions. With some machines, both indenters can be mounted on the turret and changed by a simple switch or computer command. Occasionally clean the indenter with a cotton swab and alcohol. Avoid creating static charges during cleaning. Never touch the indenter tip with your fingers as this will alter the measurements.

8.3 Place the specimen on the stage or in the stage clamps, so that the specimen surface is perpendicular to the indenter axis. A top-referenced clamping system for mounts is an excellent device for aligning the test plane perpendicular to the indenter, particularly if the back face of the mount is not parallel to the polished front surface. If clay is used on a slide, use very stiff clay and use high pressure when seating the specimen against the clay.

8.4 Focus the measuring microscope with a low power objective so that the specimen surface can be observed.

8.5 Adjust the light intensity and adjust the apertures for optimum resolution and contrast. Zero the measuring device according to the manufacturer's recommended method.

8.6 Select the area desired for hardness determination. Before applying the force, make a final focus using the measuring objective.

8.7 Adjust the tester so that the indenter is in the proper place for force application. Select the desired force.

8.8 Activate the tester so that the indenter is automatically lowered and makes contact with the specimen for the normally required time period. Then, remove the force either manually or automatically.

8.9 After the force is removed, switch to the measuring mode, and select the proper objective lens. Focus the image, adjust the light intensity if necessary, and adjust the apertures for maximum resolution and contrast.

8.10 Examine the indentation for its position relative to the desired location and for its symmetry.

8.10.1 If the indentation did not occur at the desired spot, the tester is out of alignment. Consult the manufacturer's instruction manual for the proper procedure to produce alignment. Make another indentation and recheck the indentation location. Readjust and repeat as necessary.

8.10.2 For a Knoop indentation, if one half of the long diagonal is more than 10 % longer than the other diagonal half, or if both ends of the indentation are not in sharp focus, the test specimen surface may not be perpendicular to the indenter axis. Such an indent may yield incorrect data and the calculated HK based upon it should be reported outside these limits. Check the specimen alignment and make another test to be sure that the test data is correct.

8.10.3 For a Vickers indentation, if one half of either diagonal is more than 5 % longer than the other half of that diagonal, or if the four corners of the indentation are not in sharp focus, the test surface may not be perpendicular to the indenter axis. Such an indent may yield incorrect data and the calculated HV based upon it should be reported outside these limits. Check the specimen alignment and make another test to be sure that the test data is correct.

8.10.4 If the diagonal legs are unequal as described in 8.10.2 or 8.10.3, rotate the specimen 90° and make another indentation in an untested region. If the nonsymmetrical aspect of the indentations has rotated 90°, then the specimen surface is not perpendicular to the indenter axis. If the nonsymmetrical nature of the indentation remains in the same orientation, check the indenter for misalignment or damage.

8.10.5 Some materials may have nonsymmetrical indentations even if the indenter and the specimen surface are perfectly aligned. Tests on single crystals or on textured materials may produce such results. When this occurs, check the alignment using a test specimen, such as a standard, known to produce uniformly shaped indentations.

8.10.6 Brittle materials, such as ceramics, may crack as a result of being indented. Specific details for testing ceramics are contained in Test Methods C1326 and C1327.

8.11 Measure the long diagonal of a Knoop indentation, or both diagonals of a Vickers indentation, in accordance with the manufacturer's instruction manual.

8.11.1 Determine the length of the long diagonal of a Knoop indentation or both diagonals of a Vickers indentation to within 0.1 μm (see 6.3). For the Vickers indentations, average the two diagonal length measurements.

8.12 Compute the Knoop or Vickers hardness number using the appropriate equation in Section 3 or using tables supplied with the tester, respectively. Modern testers usually give an automatic readout of the hardness after the diagonal or diagonals have been measured.

8.13 *Spacing of Indentations*—Generally, more than one indentation is made on a test specimen. It is necessary to ensure that the spacing between indentations is large enough so that adjacent tests do not interfere with each other. Because

face-centered cubic (FCC) metals (for example, austenitic stainless steels, copper, nickel, silver and gold) work harden more dramatically than body-centered cubic (BCC) metals (ferritic steels, for example), the indent spacing distance is more critical for FCC metals as the deformation zone around the indent is larger than for a BCC metal, as mentioned in 7.1.2.

8.13.1 For most testing purposes, the minimum recommended spacing between separate tests and the minimum distance between an indentation and the surface of the specimen, are illustrated in Fig. 3.

8.13.2 For some applications, closer spacing of indentations than those shown in Fig. 3 may be necessary. If a closer indentation spacing is used, it shall be the responsibility of the testing laboratory to verify the accuracy of the testing procedure. Parallel, staggered bands of indents from the surface inward can be utilized to obtain closer overall spacing of indents with respect to the distance from the surface than can be safely done with a single line of indents from the surface inward, or within the interior of the specimen.

9. Report

9.1 Report the following information:

9.1.1 The number of tests and, where appropriate or required, the mean, standard deviation and 95% confidence interval for the tests. Due to the long history of hardness calculations, and because the traditional kg/mm^2 unit is not part of the SI system, the calculated numbers will be reported without mention of the units. Also, due to the general unfamiliarity of the metallurgical community with hardness numbers in GPa, and the rather narrow range of GPa values for metals, a "soft" SI system approach is recommended.

9.1.2 Test force, and

9.1.3 Any unusual conditions encountered during the test.

9.2 The symbols HK for Knoop hardness and HV for Vickers hardness shall be used with the reported numerical values.

9.2.1 For this standard, the microindentation hardness test results can be reported in several different ways. For example, if the Knoop hardness was found to be 400, and the test force was 100 gf, the test results may be reported as follows:

9.2.1.1 For microindentation hardness tests, where the test force is generally in gram force units, with test forces ≤1000 gf, this result can be reported as 400 HK 0.1, for example, when a test at 100 gf yields a Knoop hardness of 400. The same approach is used to report the Vickers hardness.

9.2.1.2 In the SI system the hardness would be reported as 3.92 GPa, but this practice is not preferred for the reasons stated in 9.1.1.

9.2.1.3 For nonstandard dwell times, other than 10 to 15 s, the hardness would be reported as 400 HK 0.1/22 s. In this case, 22 s would be the actual time of the full load dwell time.

9.2.1.4 For macro-Vickers tests with forces >1 kgf, see Test Method E92 for the recommended notation.

9.3 Examples of the calculation of measurement uncertainty are given in Test Method E92.

10. Precision and Bias

10.1 The precision and bias of microindentation hardness measurements depend on strict adherence to the stated test procedure and are influenced by instrumental and material factors and indentation measurement errors.

10.2 The consistency of agreement for repeated tests on the same material is dependent on the homogeneity of the material, reproducibility of the hardness tester, and consistent, careful measurement of the indents by a competent operator.

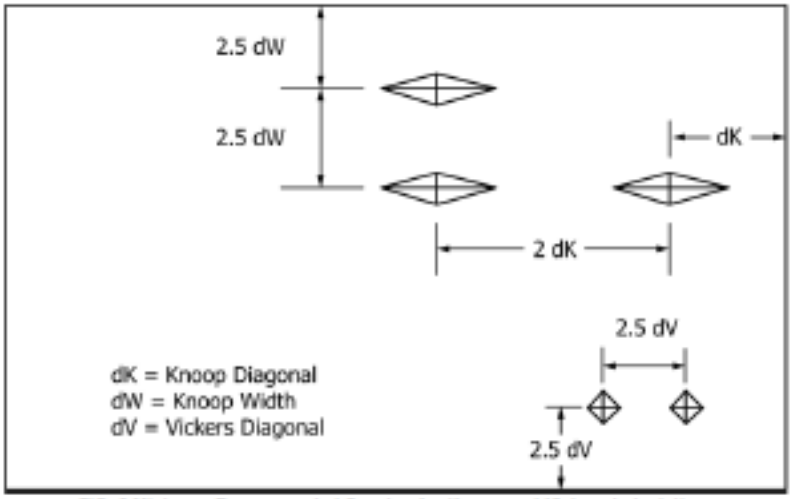


FIG. 3 Minimum Recommended Spacing for Knoop and Vickers Indentations

10.3 Instrumental factors that can affect test results include: accuracy of loading; inertia effects; speed of loading; vibrations; the angle of indentation; lateral movement of the indenter or specimen; and, indentation and indenter shape deviations.

10.3.1 Vibrations during indenting will produce larger indentations with the potential influence of vibrations becoming greater as the force decreases (2, 3).

10.3.2 The angle between the indenter and specimen surface should be within 2° of perpendicular. Greater amounts of tilting may produce non-uniform indentations and incorrect test results.

10.4 Material factors that can affect test results include: specimen homogeneity, orientation or texture effects; improper specimen preparation; low specimen surface reflectivity; and, transparency of the specimen.

10.4.1 Residual deformation from mechanical polishing must be removed, particularly for low-force (≤200 gf) testing.

10.4.2 Distortion of the indentation shape, due to either crystallographic or microstructural texture, influences diagonal lengths and the validity of the calculated hardness.

10.4.3 Plastic deformation during indentation can produce ridging around the indentation periphery that will affect diagonal measurement accuracy.

10.4.4 Testing of etched surfaces, depending on the extent of etching, may produce results that are different from those obtained on unetched surfaces (2).

10.5 Measurement errors that can affect test results include: inaccurate calibration of the measuring device; inadequate resolving power of the objective; insufficient magnification; operator bias in sizing the indentations; poor image contrast; non-uniform illumination; and, improper zeroing of the measuring device.

10.5.1 The accuracy of microindentation hardness testing is strongly influenced by the accuracy to which the indentations can be measured.

10.5.2 The error in measuring the diagonals increases as the numerical aperture of the measuring objective decreases (4, 5). In general, indents <30 µm in length should be measured with objectives having greater magnification than 40 or 50×. Image contrast between the indent and the specimen is critical for precise measurement of diagonal length.

10.5.3 Bias is introduced if the operator consistently undersizes or over-sizes the indentations.

10.6 Some of the factors that affect test results produce systematic errors that influence all test results while others primarily influence low-force (≤25 gf) test results (6). Some of these problems occur continually, others may occur in an undefined, sporadic manner. Low-force hardness tests are influenced by these factors to a greater extent than higher force tests.

10.7 For both the Vickers and Knoop hardness tests, the calculated microindentation hardness is a function of three variables: force, indenter geometry and diagonal measurement. For the Vickers test, the error in measuring the diagonals has a bigger effect on the precision of the HV value than a larger error in the test force or the face geometry. For the Knoop test, an error in measuring the long diagonal has a bigger influence on the precision of the HK value than a larger error in the test force. But, errors in the two face angles, Fig. 1, have a very significant effect on the precision of the HK value.

10.8 Three separate interlaboratory studies have been conducted in accordance with Practice E691 to determine the precision, repeatability, and reproducibility of this test method. The three studies are defined as follows: *(a)* Knoop and Vickers tests, six test forces in the micro range, twelve laboratories, manual measurements, and seven different hardness level specimens (see 10.8.1 and Appendix X1). Results were published in 1989 (7, 8) and in ASTM Research Report RR:E04-1004.[5] *(b)* Knoop and Vickers tests, two test forces in the micro range, seven laboratories, image analysis and manual measurements, four different hardness level specimens (see 10.8.2, Appendix X2 and ASTM Research Report RR:E04-1006).[6] *(c)* Knoop and Vickers tests, six test forces in the micro range, twenty-five laboratories, manual measurements, six different hardness level specimens (see 10.8.3, Appendix X3 and ASTM Research Report RR:E04-1007).[7]

10.8.1 An interlaboratory test program was conducted in accordance with Practice E691 to develop information regarding the precision, repeatability, and reproducibility of the measurement of Knoop and Vickers indentations (supporting data have been filed at ASTM Headquarters; request RR:E04-1004).[5] The test forces were 25, 50, 100, 200, 500, and 1000 gf on three ferrous and four nonferrous specimens (7, 8). Twelve laboratories measured the indentations, five of each type at each force on each sample. Additional details of this study are given in Appendix X1.

10.8.1.1 Tests of the three ferrous specimens revealed that nine laboratories produced similar measurements while two laboratories consistently undersized the indentations and one laboratory consistently oversized the indentations; that is, biased results were produced. These latter results were most pronounced as the force decreased and specimen hardness increased (that is, as the diagonal size decreased) and were observed for both Vickers and Knoop indentations. Results for the lower hardness nonferrous indentations produced better agreement. However, none of the laboratories that obtained higher or lower results on the ferrous specimens measured the nonferrous indentations.

10.8.1.2 *Repeatability Interval*—The difference due to test error between two test results in the same laboratory on the same material increases with increasing specimen hardness and with decreasing test force (see X1.4.4).

10.8.1.3 *Reproducibility Interval*—The difference in test results on the same material tested in different laboratories increased with increasing specimen hardness and with decreasing test force (see X1.4.5).

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E04-1004. Contact ASTM Customer Service at service@astm.org.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E04-1006. Contact ASTM Customer Service at service@astm.org.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E04-1007. Contact ASTM Customer Service at service@astm.org.

10.8.1.4 The within-laboratory and between-laboratory precision values improved as specimen hardness decreased and test force increased. The repeatability interval and reproducibility interval were generally larger than the precision estimate, particularly at low test forces and high specimen hardness.

10.8.2 An interlaboratory test program was conducted in accordance with Practice E691 to develop information regarding the repeatability and reproducibility of Knoop and Vickers measurements made with automated image analysis systems compared to measurements by manual procedures. Four ferrous specimens were used in the round robin. The tests were conducted at 100 gf and 300 gf. The participants in the test program measured the same indentations on the four specimens. Seven labs measured the specimens using both procedures. The Knoop indentations on specimen C1 were too long for accurate measurements to be made by one lab; hence, only six sets of measurements were made on this specimen. Near the end of the test program, specimen B1 was lost in shipping; thus only six sets of measurements were made on this specimen. Additional details of the study are contained in Appendix X2.

10.8.2.1 Repeatability concerns the variability between individual test results obtained within a single laboratory by a single operator with a specific set of test apparatus. For both the manual and automated measurements, the repeatability interval increased with specimen hardness and decreasing test force, Appendix X2. For equivalent testing conditions, the repeatability interval for automated measurements was slightly larger than for manual measurements.

10.8.2.2 Reproducibility deals with the variability between single test results obtained by different laboratories applying the same test methods to the same or similar test specimens. For both the manual and automated measurements, the reproducibility interval increased with specimen hardness and decreasing test force, Appendix X2. For equivalent testing conditions, the reproducibility interval for automated measurements was slightly larger than for manual measurements.

10.8.2.3 Neither Practice E691, nor any other ASTM standard, deals with comparing test results of a single property made by two different test methods. Hence, it is not possible to statistically and accurately compare the hardness measurements made by the manual and automated procedures. However, this information is graphically represented for comparative purposes, X2.6.

10.8.3 Tests of six ferrous alloys with hardness values of <20 HRC, 30, 40, 50, 60 and 67 HRC were tested using Knoop and Vickers tests at a variety of test forces, usually 25, 50, 100, 300, 500 and 1000 gf (except that the lowest test forces for Vickers tests of the 60 and 67 HRC specimens were not performed). Twenty-five different laboratories tested the steels using the Vickers test while thirteen different laboratories tested the steels using the Knoop test. Additional details of this study are given in Appendix X3.

10.8.3.1 Repeatability and reproducibility statistics were determined for the Knoop and Vickers diagonal measurements. Results are tabulated in Table X3.1 and Table X3.2 and are shown graphically in Fig. X3.1 and Fig. X3.2.

10.8.3.2 Repeatability and reproducibility statistics were determined for the Knoop and Vickers hardness values. Results are tabulated in Table X3.3 and Table X3.4 and are shown graphically in Fig. X3.3 and Fig. X3.4.

11. Conversion to Other Hardness Scales or Tensile Strength Values

11.1 There is no generally accepted method for precise conversion of Knoop or Vickers microindentation hardness numbers to other hardness scales or tensile strength values. Such conversions are empirical and are limited in precision and should be used with caution, except for special cases where a reliable basis for the conversion has been obtained by comparison tests. For loads ≥ 25 gf microindentation Vickers hardness numbers are in statistical agreement with macro-Vickers hardness numbers. Refer to Standard Hardness Conversion Tables in E140.

12. Keywords

12.1 hardness; indentation; Knoop; microindentation; Vickers

ANNEXES

(Mandatory Information)

A1. VERIFICATION OF KNOOP AND VICKERS HARDNESS TESTING MACHINES AND INDENTERS

A1.1 Scope

A1.1.1 Annex A1 specifies three types of procedures for verifying microindentation (Knoop and Vickers) hardness testing machines: direct verification, indirect verification, and periodic verification. This annex also contains geometric specifications for the indenter. A control chart method for monitoring the consistency of microindentation measurements based on the periodic verification tests and detecting measurement deviations is described in Practices E2554 and E2587.

A1.1.2 Direct verification is a process normally performed by the manufacture for verifying that critical components of the hardness testing machine are within allowable tolerances by direct measurement of the applied test forces, the indentation measuring system, and the testing cycle. For additional information about direct verification see Test Method E92.

A1.1.3 Indirect verification is a process performed by the user of the machine, or by an outside certification agency, to periodically verify the performance of the testing machine by

means of standardized test blocks. For additional information about the indirect verification procedure, see Test Method E92.

A1.1.4 The periodic (formerly called "weekly") verification is a process for monitoring the performance of the testing machine between indirect verifications by means of standardized test blocks and is performed by the user.

A1.2 General Requirements

A1.2.1 The testing machine shall be verified at specific instances and at periodic intervals as specified in Table A1.1, and when circumstances occur that may affect the performance of the testing machine. See Annex A1 in Test Method E92 for interval details for direct and indirect verifications.

A1.2.2 All instruments used to make measurements required by this Annex shall be calibrated traceable to national standards when a system of traceability exists, except as noted otherwise.

A1.2.3 Periodic verification and the indirect verification of the testing machine shall be performed at the location where the tester is used.

A1.2.4 Direct verification of newly manufactured or rebuilt testing machines may be performed at the place of manufacture, rebuild or the location of use. Details of this procedure can be found in Test Method E92.

NOTE A1.1—It is recommended that the calibration agency that is used to conduct the verifications of microindentation hardness testing machines be accredited to the requirements of ISO 17025 (or an equivalent) by a recognized accrediting body that operates to the requirements of ISO Guide 58.

A1.2.5 *Verification of Indenter*—The geometry of the indenter is verified at the time of manufacturing and it is mandatory for new machines. Subsequent verifications of the indenter are performed by visual inspection of the resulting indentation; it is usually sufficient for the user to verify the absence of defects from the shape of indentations performed on test blocks. Details of this process are given in Test MethodE92.

A1.3 Periodic Verification

A1.3.1 The periodic (formerly known as the "weekly") verification is intended as a tool for the user to monitor the performance of the testing machine between indirect verifications. At a minimum, the periodic verification shall be performed in accordance with the schedule given in Table A1.1 for each microindentation hardness indenter that will be used.

A1.3.2 It is recommended that the periodic verification procedures be performed whenever the indenter is changed, that is, if one indenter is physically removed from the port and another is inserted into its place. This is not required with machines that have both types of indenter mounted on the same turret. It is also recommended to perform a periodic verification when loads are changed (to verify that the load is not "hanging up").

A1.3.3 *Periodic Verification Procedures*—The procedure to use when performing a periodic verification is as follows.

A1.3.3.1 At least one standardized test block that meets the requirements of Annex A2 shall be used for each microindentation hardness indenter to be used. When test blocks are commercially available, the hardness level of the test blocks shall be chosen at approximately the same hardness value as the material to be measured. If various hardness ranges are to be made, it is recommended to take a test block from each range of hardness as described in Table A1.2.

A1.3.3.2 The indenter to be used for the periodic verification shall be the indenter that is normally used for testing.

A1.3.3.3 Before performing the periodic verification tests, ensure that the testing machine is working freely, the stage and test block are clean, and the measuring device is properly adjusted and zeroed.

A1.3.3.4 Make at least three hardness measurements on each of the verification test blocks. The tests shall be distributed uniformly over the surface of the test blocks.

A1.3.3.5 Let $\bar{d}$ be the average of the measurements. Determine the error E and the repeatability R in the performance of the testing machine using Eq 10 and Eq 11 from 3.4 for each standardized test block that is measured.

(1) If the error E and the repeatability R calculated for each test block is within the tolerances given in Table A1.3, the testing machine with the indenter may be regarded as performing satisfactorily.

(2) If the error E and the repeatability R calculated for any of the test blocks is outside the tolerances, the periodic verification may be repeated with a different indenter. If the average of the hardness measurements again falls outside of tolerances for any of the test blocks, an indirect verification shall be performed.

A1.3.3.6 If a testing machine fails a periodic verification, the hardness tests made since the last valid periodic verification may be suspect.

NOTE A1.2—It is highly recommended that the results obtained from the periodic verification testing be recorded using accepted Statistical Process Control techniques, such as, but not limited to, *X*-bar (measurement averages) and *R*-charts (measurement ranges), and histograms (see Practices E2554 and E2587).

A1.4 Verification Report

A1.4.1 A verification report is required for direct and indirect verifications. A verification report is not required for a periodic verification. Additional details concerning creation of the verification report can be found in Test Method E92.

TABLE A1.1 Verification Schedule for a Microindentation Hardness Testing Machine

Verification Procedure	Schedule
Periodic Verification	Required each week that the machine is used. Recommended whenever the indenter is physically removed and replaced by another indenter.

TABLE A1.2 Hardness Ranges Used for Periodic Verification

Range	Knoop	Vickers
Low	< 250	< 240
Mid	250–650	240–600
High	> 650	> 600

TABLE A1.3 Repeatability and Error of Test Machines— Periodic Verification by Standardized Test Blocks Based on Measured Diagonal Lengths[A]

Hardness Range of Standardized Test Blocks		Force, gf	R Maximum Repeatability (%)	E Maximum Error (%)
Knoop	Vickers			
HK > 0	HV > 0	1 ≤ F <100	13	3
HK < 100	HV < 100	100 ≤ F ≤ 1000	13	3
100 ≤ HK ≤ 250	100 ≤ HV ≤ 240	100 ≤ F < 500	13	2
250 < HK ≤ 650	240 < HV ≤ 600		5	2
HK > 650	HV > 600		4	2
100 ≤ HK ≤ 250	100 ≤ HV ≤ 240	500 ≤ F ≤ 1000	8	2
250 < HK ≤ 650	240 < HV ≤ 600		4	2
HK > 650	HV > 600		3	2

[A] In all cases, the repeatability is the greater of the percentage given or 1 μm; the maximum error is the greater of the value obtained or 0.5 μm.

A1.4.2 The verification report shall be produced by the person performing the verification and include the following information when available as a result of the verification performed.

A1.4.2.1 Full details of the verification report can be found in Test Method E92.

A1.4.2.2 The basic components of the verification report, as defined in detail in Test Method E92, are summarized below.

(1) Identification of the hardness testing machine and the indenters used.

(2) Means of verification (test blocks, elastic proving devices, etc.) with statements defining traceability to a national standard.

(3) The microindentation hardness scale(s) verified.

(4) The individual or calculated results used to determine whether the testing machine meets the requirements of the verification performed. Measurements made to determine the as-found condition of the testing machine shall be included whenever they are made.

(5) Description of adjustments or maintenance done to the testing machine.

(6) Date of verification and reference to the verifying agency or department.

(7) Signature of the person performing the verification.

A2. CALIBRATION OF STANDARDIZED HARDNESS TEST BLOCKS FOR MICROINDENTATION HARDNESS TEST MACHINES

A2.1 Scope

A2.1.1 The calibration of standardized hardness test blocks used to verify microindentation hardness test machines is described in Test Method E92. The standardizing machine shall meet the direct verification method described in Test Method E92.

A2.1.2 Re-polishing of the test block will invalidate the standardization and is not recommended. Cleaning of the polished test block surface is often required in normal usage but must not alter the hardness or quality of the polished test surface.

A2.2 Certification of Standardized Test Block

A2.2.1 The certificate accompanying each standardized hardness test block shall include the following information: the arithmetic mean of each group of five impression diagonals; the arithmetic mean and standard deviation of all impression diagonals, the corresponding hardness value, the test force, serial number of the test block, name of the manufacturer and certifying organization, magnification used, and the date.

APPENDIXES

(Nonmandatory Information)

X1. RESULTS OF INTERLABORATORY TEST OF THE MEASUREMENT OF MICROINDENTATIONS

X1.1 Introduction

X1.1.1 This interlaboratory test program (7, 8) was conducted to develop precision and bias estimates for the measurement of both Knoop and Vickers indentations using forces of 25 to 1000 gf for ferrous and nonferrous specimens covering a wide range of hardness (see Research Report RR:E04-1004).[5]

X1.2 Scope

X1.2.1 This interlaboratory test program provides information on the measurement of the same indentations by different laboratories according to the procedures of Practice E691.

X1.3 Procedure

X1.3.1 Five indentations were made under controlled conditions at each force (25, 50, 100, 200, 500, and 1000 gf), with both Knoop and Vickers indenters using three ferrous and four nonferrous specimens.

X1.3.2 Twelve laboratories measured the indentations on the ferrous specimens and the nonferrous specimens. Two laboratories measured the hardness of both groups.

X1.3.3 Each laboratory used the same stage micrometer to calibrate their measuring device.

X1.3.4 Results were tabulated and analyzed in accordance with Practice E691.

X1.4 Results

X1.4.1 For the three ferrous specimens, results from nine laboratories showed general agreement as to the diagonal sizes. Two other laboratories consistently undersized the indentations (higher hardness) and one laboratory consistently oversized the indentations (lower hardness). This bias was observed with both Vickers and Knoop indentations sized by these laboratories with the degree of bias increasing as the indentation size decreased and the specimen hardness increased. Test on the four nonferrous specimens produced general agreement, but none of the three laboratories that produced biased results for the ferrous specimens measured the nonferrous specimens.

X1.4.2 For the Vickers test data, the calculated hardness increased with increasing force and then became reasonably constant. This trend was apparent in the data from the nine consistent laboratories (ferrous specimens) and for the laboratory that oversized the indentations. The two laboratories that consistently undersized the Vickers indentations exhibited substantial data scatter for the tests with forces of less than 100 gf. However for higher forces, their indentation measurements were relatively constant. The force at which the hardness became relatively constant increased with increasing specimen hardness. For specimens below about 300 HV, there was relatively little difference in HV over the test force range.

X1.4.3 For the Knoop test data, most of the laboratories agreed that the hardness decreased continually with increasing test force and then became reasonably constant. However, the two laboratories that exhibited outlier data for the ferrous specimens did show the opposite trend; this is highly unusual. The difference in HK values between low forces and high forces increased with increasing specimen hardness. For specimens with hardness below about 300 HK, the difference in hardness was quite small over the test force range.

X1.4.4 *Repeatability Interval*—The difference due to test error between two test results in the laboratory on the same material was calculated using the $(S_r)j$ values, the pooled within-laboratory standard deviation. $(S_r)j$ increased with diagonal size and the relationship varied for each material and test type. Table X1.1 lists regression equations that show the relationship between $(S_r)j$ and the diagonal length, μm. The repeatability interval $(I_r)j$, was calculated based on the relationships in Table X1.1. Because the repeatability intervals are also a function of diagonal length, regression equations were also calculated, Table X1.2. The repeatability intervals, in terms of Knoop and Vickers values for ferrous and nonferrous specimens, are shown in Figs. X1.1-X1.4.

X1.4.5 *Reproducibility Interval*—The difference in test results on the same material in different laboratories was calculated using the $(S_R)j$ values, the between-laboratory estimate of precision. $(S_R)j$ increased with diagonal size and the relationship varied for each material and test type. Table X1.3 lists the regression equations that show the relationship between $(S_R)j$ and the diagonal length, μm. The reproducibility intervals $(I_R)j$, were calculated based on the relationships shown in Table X1.3. Because the reproducibility intervals are also a function of diagonal length, regression equations were also calculated, Table X1.4. The reproducibility intervals, in terms of Knoop and Vickers values for the ferrous and nonferrous specimens, are shown in Figs. X1.1-X1.4.

X1.4.6 The within-laboratory and between-laboratory precision values were calculated from $(V_r(\%))j$ and $(V_L(\%))j$ which are the coefficients of variation for within-laboratory and between-laboratory tests. Both are a function of the length of the diagonal. The within-laboratory and between-laboratory precision values were relatively similar for both Vickers and Knoop test data, either ferrous or nonferrous. In general, the repeatability intervals and reproducibility intervals were larger than the precision estimates, particularly at low test forces and high specimen hardness.

TABLE X1.1 Relationship Between Diagonal Length and (S_{ij}), the Pooled Within-Laboratory Standard Deviation

Material	Test	Regression Equation	Correlation Coefficient
Ferrous	Vickers	$(S_{ij}) = 0.231 + 0.00284\ d_i$	0.535
Ferrous	Knoop	$(S_{ij}) = 0.216 + 0.006\ d_i$	0.503
Nonferrous	Vickers	$(S_{ij}) = 0.373 + 0.008\ d_i$	0.662
Nonferrous	Knoop	$(S_{ij}) = 0.057 + 0.0177\ d_i$	0.8196

TABLE X1.2 Relationship Between the Diagonal Length and (I_{ij}), the Repeatability Interval

Material	Test	Regression Equation
Ferrous	Vickers	$(I_{ij}) = 0.653 + 0.008\ d_i$
Ferrous	Knoop	$(I_{ij}) = 0.614 + 0.017\ d_i$
Nonferrous	Vickers	$(I_{ij}) = 1.0556 + 0.0226\ d_i$
Nonferrous	Knoop	$(I_{ij}) = 0.161 + 0.05\ d_i$

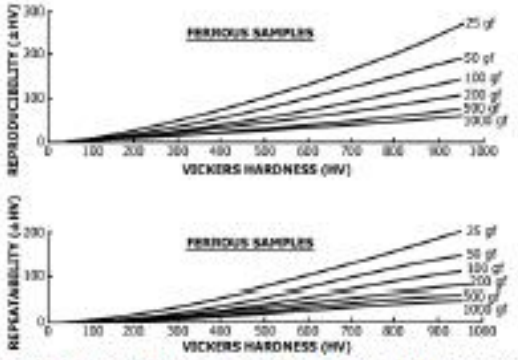


FIG. X1.1 Repeatability and Reproducibility Intervals in Terms of Vickers Hardness (±) for the Ferrous Sample as a Function of Test Load and Specimen Hardness

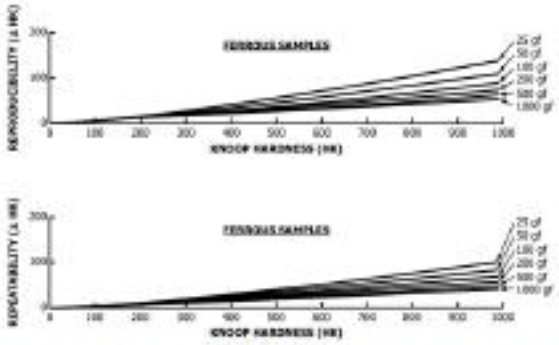
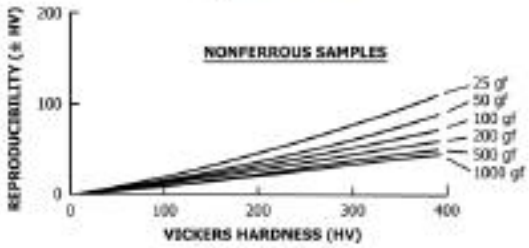


FIG. X1.2 Repeatability and Reproducibility Intervals in Terms of Knoop Hardness (±) for the Ferrous Samples as a Function of Test Load and Specimen Hardness



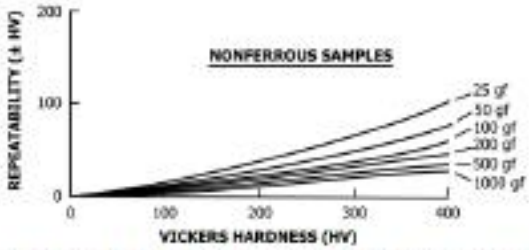
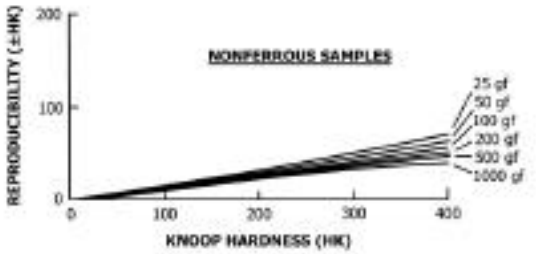


FIG. X1.3 Repeatability and Reproducibility Intervals in Terms of Vickers Hardness (±) for the Nonferrous Samples as a Function of Test Load and Specimen Hardness



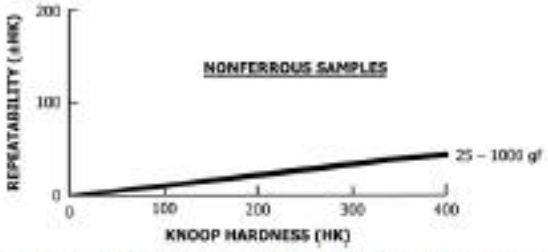


FIG. X1.4 Repeatability and Reproducibility Intervals in Terms of Knoop Hardness (±) for the Nonferrous Samples as a Function of Test Load and Specimen Hardness

TABLE X1.3 Relationship Between Diagonal Length and (S_R), the Between-Laboratory Estimate of Precision

Material	Test	Regression Equation	Correlation Coefficient
Ferrous	Vickers	$(S_R) = 0.31 + 0.004\ d_1$	0.747
Ferrous	Knoop	$(S_R) = 0.333 + 0.007\ d_1$	0.899
Nonferrous	Vickers	$(S_R) = 0.357 + 0.0156\ d_1$	0.8906
Nonferrous	Knoop	$(S_R) = 0.378 + 0.0177\ d_1$	0.8616

TABLE X1.4 Relationship Between the Diagonal Length and (I_R), the Repeatability Interval

Material	Test	Regression Equation
Ferrous	Vickers	$(I_R) = 0.877 + 0.0113\ d_1$
Ferrous	Knoop	$(I_R) = 0.946 + 0.0198\ d_1$
Nonferrous	Vickers	$(I_R) = 1.0103 + 0.0441\ d_1$
Nonferrous	Knoop	$(I_R) = 1.07 + 0.05\ d_1$

X2. RESULTS OF AN INTERLABORATORY TEST COMPARING MICROINDENTATION HARDNESS TESTING USING MANUAL AND AUTOMATED MEASURING SYSTEMS

X2.1 Introduction

X2.1.1 An interlaboratory test program was conducted to develop information comparing Knoop and Vickers microindentation hardness tests made with measurements using automated image analysis systems and by the standard manual procedure. Four ferrous specimens were used in the test program (see Research Report RR:E04-1006).[6]

X2.2 Scope

X2.2.1 This interlaboratory test program provides information on measurements of the same indentations made by different laboratories using two different measuring methods according to the procedures of Practice E691.

X2.3 Procedure

X2.3.1 The test was conducted under controlled conditions using loads of 100 gf and 300 gf. Ten Knoop and ten Vickers indentations were made for each load, a total of 40 indentations. The participants in the test program measured the same indentations on the four specimens. Seven laboratories measured the specimens using both procedures. The results of these seven sets of measurements were used for the analysis. The Knoop indentations on specimen C1 were too long for accurate measurements to be made by one lab; hence, only six sets of measurements were made on this specimen. Near the end of the test program, specimen B1 was lost in shipping; thus only six sets of measurements were made on this specimen.

X2.4 Repeatability

X2.4.1 Repeatability concerns the variability between individual test results obtained within a single laboratory by a single operator with a specific set of test apparatus. For both the manual and automated measurements, the repeatability interval increased with specimen hardness and decreasing test force, Tables X2.1-X2.4, and Figs. X2.1-X2.4. For equivalent testing conditions, the repeatability interval for automated measurements was slightly larger than for manual measurements.

X2.5 Reproducibility

X2.5.1 Reproducibility deals with the variability between single test results obtained by different laboratories applying the same test methods to the same or similar test specimens. For both the manual and automated measurements, the reproducibility interval increased with specimen hardness and decreasing test force, Tables X2.1-X2.4, and Figs. X2.1-X2.4. For equivalent testing conditions, the reproducibility interval for automated measurements was slightly larger than for manual measurements.

X2.6 Comparisons

X2.6.1 Neither Practice E691, nor any other ASTM standard, deals with comparing test results of a single property made by two different test methods. Hence, it is not possible to statistically and accurately compare the hardness measurements made by the manual and automated procedures. However, this information is graphically represented for comparative purposes, Figs. X2.5-X2.8.

TABLE X2.1 Precision Statistics for Manual and Automated Knoop Tests at 100 gf Load

Manual							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	7	228.62	6.88	9.30	11.16	26.03	31.32
D1	7	344.80	10.54	9.80	14.06	27.44	39.36
A2	7	491.48	28.67	14.87	31.95	41.63	89.45
B1	6	901.67	62.40	21.17	65.55	59.28	183.55
Automated							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	7	232.07	7.29	9.54	11.62	26.72	32.55
D1	7	348.97	10.74	9.54	14.04	26.70	39.32
A2	7	510.13	30.35	19.53	35.56	54.69	99.56
B1	6	914.72	57.82	29.22	64.13	81.83	179.56

TABLE X2.2 Precision Statistics for Manual and Automated Knoop Tests at 300 gf Load

Manual							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	6	215.81	5.49	7.66	9.10	21.44	25.49
D1	7	330.64	6.99	7.49	9.97	20.96	27.92
A2	7	466.95	17.99	11.45	21.02	32.06	58.85
B1	6	827.47	20.41	16.13	25.51	45.16	71.43
Automated							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	6	217.82	5.73	6.87	8.68	19.24	24.31
D1	7	335.76	12.23	8.22	14.50	23.03	40.61
A2	7	476.97	23.46	10.56	25.51	29.58	71.44
B1	6	821.00	24.62	10.89	26.70	30.50	74.76

TABLE X2.3 Precision Statistics for Manual and Automated Vickers Tests at 100 gf Load

Manual							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	7	205.31	6.36	6.82	9.07	19.10	25.40
D1	7	299.52	6.07	7.65	9.46	21.43	26.50
A2	7	462.76	21.58	12.29	24.53	34.42	68.69
B1	6	821.56	46.01	24.02	51.35	67.25	143.77
Automated							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	7	203.30	6.94	6.47	9.27	18.12	25.95
D1	7	299.78	14.36	5.23	15.19	14.63	42.54
A2	7	462.86	32.07	16.50	35.69	46.19	99.93
B1	6	808.17	47.72	21.30	51.82	59.63	145.09

TABLE X2.4 Precision Statistics for Manual and Automated Vickers Tests at 300 gf Load

Manual							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	7	197.07	3.40	5.32	6.09	14.91	17.06
D1	7	298.91	5.47	7.38	8.89	20.66	24.89
A2	7	474.58	18.00	12.45	21.53	34.86	60.28
B1	6	810.60	29.67	16.50	33.55	46.21	93.94
Automated							
Spec.	Labs	Mean	Sx	Sr	SR	r	R
C1	7	196.37	6.44	5.57	8.33	15.60	23.32
D1	7	297.86	10.42	6.69	12.20	18.72	34.15
A2	7	483.72	18.96	12.30	22.26	34.44	62.34
B1	6	809.55	20.55	11.60	23.31	32.49	65.27

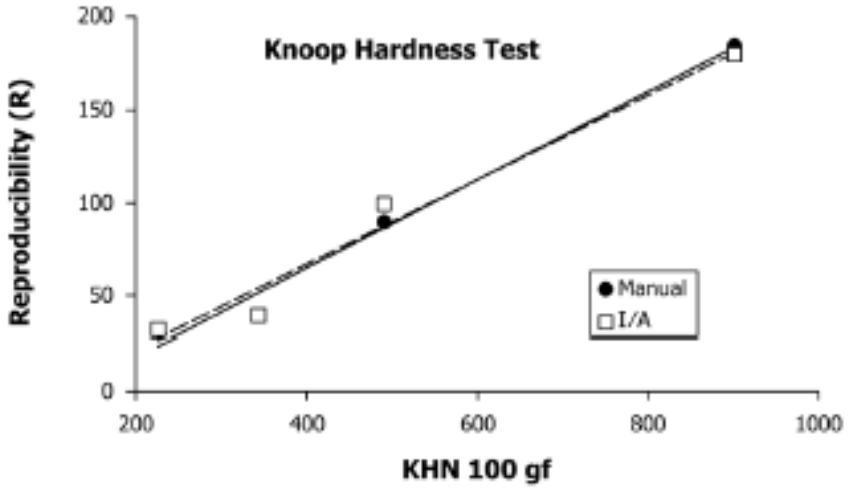


FIG. X2.1 Reproducibility of the Knoop 100 gf Manual and Automated Microindentation Hardness Tests

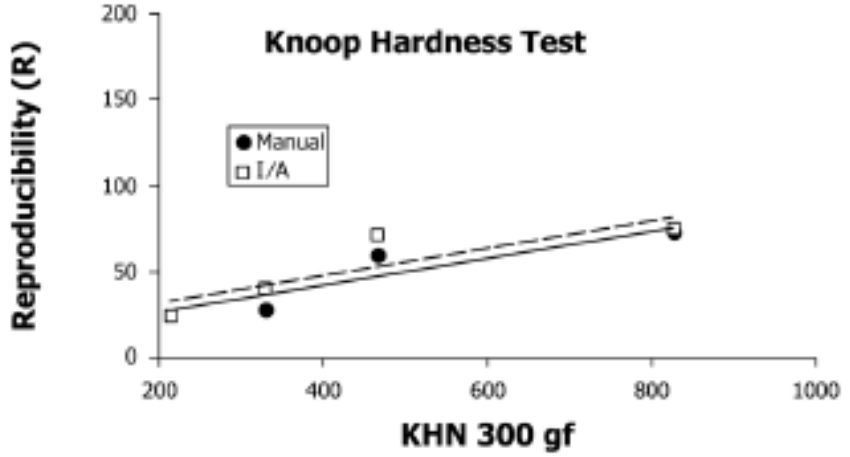


FIG. X2.2 Reproducibility of the Knoop 300 gf Manual and Automated Microindentation Hardness Tests

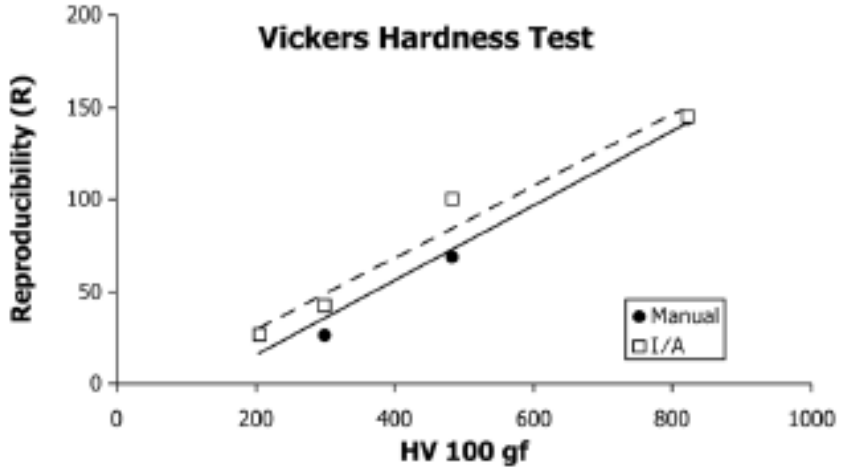


FIG. X2.3 Reproducibility of the Vickers 100 gf Manual and Automated Microindentation Hardness Tests

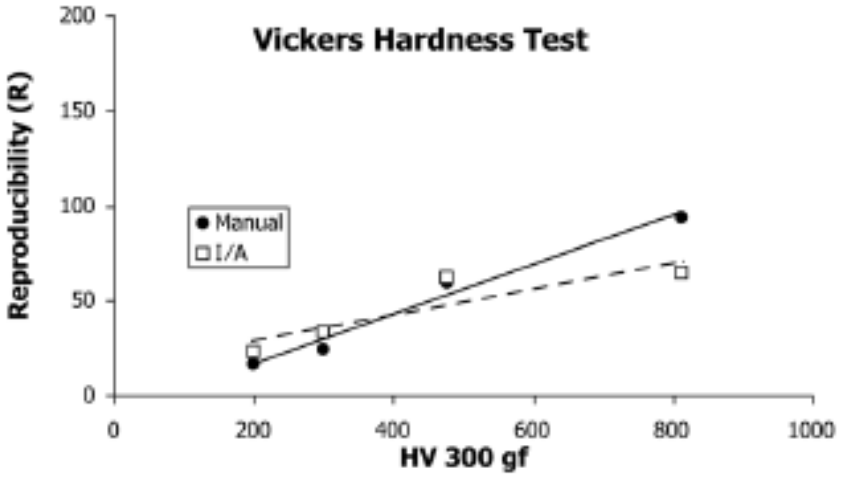


FIG. X2.4 Reproducibility of the Vickers 300 gf Manual and Automated Microindentation Hardness Tests

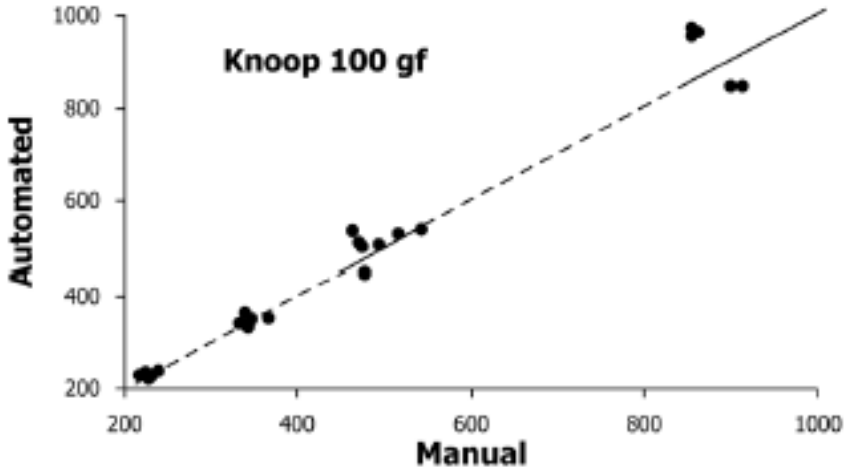


FIG. X2.5 Comparison between Knoop 100 gf Manual and Automated Microindentation Hardness Tests

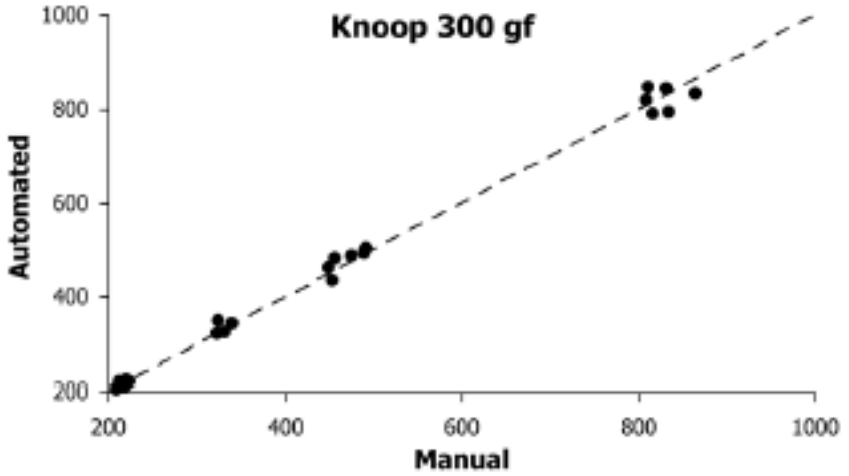


FIG. X2.6 Comparison between Knoop 300 gf Manual and Automated Microindentation Hardness Tests

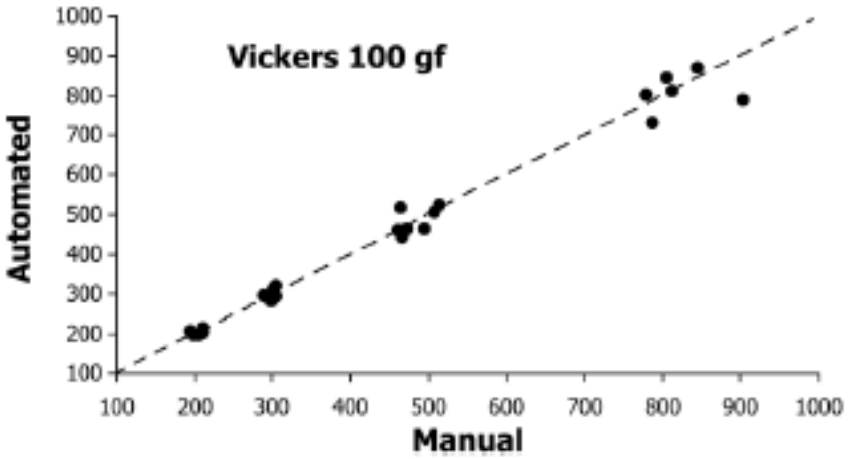


FIG. X2.7 Comparison between Vickers 100 gf Manual and Automated Microindentation Hardness Tests

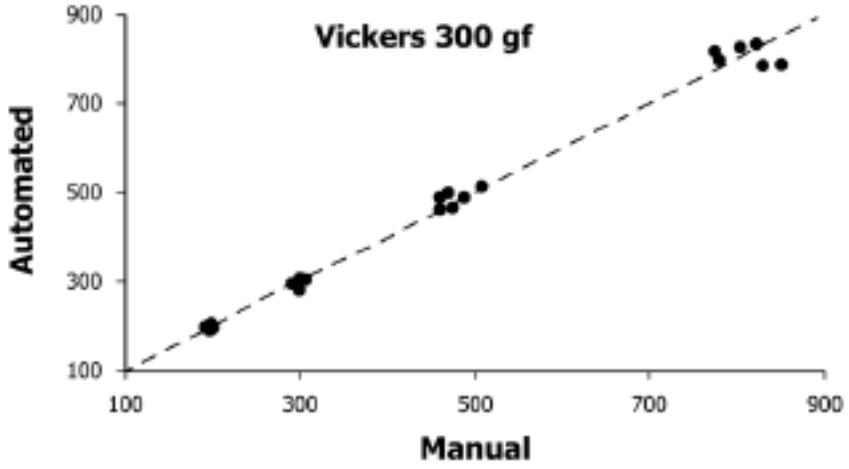


FIG. X2.8 Comparison between Vickers 300 gf Manual and Automated Microindentation Hardness Tests

X3. RESULTS OF INTERLABORATORY TEST OF THE MEASUREMENT OF MICROINDENTATIONS

X3.1 Introduction

X3.1.1 The interlaboratory program was conducted on steels to develop precision statistics for Knoop and Vickers tests (see Research Report RR:E04-1007).[7]

X3.2 Scope

X3.2.1 Twenty five laboratories tested six steel specimens for Vickers hardness and thirteen laboratories tested the six steel specimens for Knoop hardness, all as a function of test forces ranging from 25 to 1000 gf, except for the hardest specimens.

X3.2.2 The precision statement was determined through statistical examination of results from twenty-five laboratories, on six ferrous materials. These six ferrous materials were described as:

Specimen A: H13, mill annealed, hardness less than 20 HRC
Specimen B: H13, austenitized, quenched, and tempered to ~ 50 HRC
Specimen C: H13, austenitized, quenched, and tempered to ~ 40 HRC
Specimen D: H13, austenitized, quenched, and tempered to ~ 30 HRC
Specimen E: O1, austenitized, quenched and tempered O1 steel to ~ 60 HRC
Specimen F: T15, PM, austenitized, quenched and tempered to ~ 67 HRC

NOTE X3.1—To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

X3.3 Results

X3.3.1 Details of this study can be obtained from ASTM; request Research Report RR:E04-1006.[6]

X3.3.2 *Repeatability limit (r)*—Two test results obtained within one laboratory were judged not equivalent if they differed by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

X3.3.3 Repeatability limits in diagonal lengths (μm) are listed Table X3.1 and Table X3.2 and in hardness units (HK, HV) in Table X3.3 and Table X3.4.

X3.3.4 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

X3.3.5 Reproducibility limits in diagonal lengths (μm) are listed in Table X3.1 and Table X3.2 and Fig. X3.1 and Fig. X3.2 and in hardness units (HK, HV) in Table X3.3 and Table X3.4 and Fig. X3.3 and Fig. X3.4.

X3.3.6 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

X3.3.7 Any judgment in accordance with statements X3.3.2 and X3.3.4 would have an approximate 95% probability of being correct.

X3.3.8 The data are listed in Tables X3.1-X3.4 and are shown graphically in Figs. X3.1-X3.4.

TABLE X3.2 Precision Statistics for an Interlaboratory Study of the Knoop Microindentation Hardness Test for Ferrous Specimens in Diagonal Units (μm)

Specimen	Test Force (gf)	Average Diagonal (μm) $\bar{d}$	Standard Deviation (μm) S_d	Repeatability Standard Deviation (μm) S_r	Reproducibility Standard Deviation (μm) S_R	Repeatability Limit (μm) r	Reproducibility Limit (μm) R
A	25	35.61	1.40	0.72	1.54	2.00	4.31
	50	51.77	1.33	1.11	1.66	3.12	4.66
	100	74.84	1.65	1.77	2.28	4.95	6.40
	300	132.28	2.63	2.57	3.50	7.20	9.79
	500	171.51	2.07	2.46	3.02	6.89	8.45
	1000	243.11	1.72	2.96	3.16	8.29	8.84
B	25	23.66	0.95	0.48	1.04	1.34	2.91
	50	34.33	0.94	0.56	1.07	1.57	2.99
	100	49.61	1.12	0.65	1.26	1.82	3.54
	300	88.64	1.39	0.88	1.59	2.45	4.46
	500	115.48	1.68	1.11	1.95	3.11	5.46
	1000	164.38	1.65	1.52	2.14	4.25	5.98
C	25	27.62	1.33	0.49	1.41	1.38	3.93
	50	39.47	1.14	0.50	1.22	1.39	3.43
	100	56.66	1.05	0.64	1.20	1.79	3.35
	300	100.14	1.25	0.81	1.44	2.26	4.03
	500	130.19	1.50	0.83	1.68	2.33	4.69
	1000	184.84	1.79	1.19	2.08	3.33	5.82
D	25	31.04	1.04	0.46	1.11	1.28	3.12
	50	44.64	0.85	0.46	0.95	1.30	2.65
	100	64.22	1.06	0.67	1.24	1.89	3.47
	300	113.94	0.94	0.82	1.19	2.29	3.33
	500	148.16	1.16	0.74	1.33	2.06	3.73
	1000	210.10	2.03	1.64	2.50	4.58	7.00
E	25	20.02	0.72	0.48	0.84	1.36	2.34
	50	29.03	1.00	0.48	1.09	1.34	3.05
	100	42.21	1.15	0.52	1.24	1.46	3.46
	300	76.03	1.00	0.53	1.11	1.48	3.10
	500	99.25	1.06	0.49	1.15	1.37	3.21
	1000	141.67	1.27	0.85	1.48	2.39	4.15
T	25	17.14	0.88	0.48	0.98	1.35	2.76
	50	25.59	1.03	0.47	1.12	1.32	3.12
	100	37.20	1.45	0.52	1.52	1.46	4.26
	300	67.43	1.39	0.65	1.51	1.82	4.22
	500	88.27	1.11	0.66	1.26	1.86	3.53
	1000	126.96	1.47	0.75	1.61	2.09	4.52

TABLE X3.3 Precision statistics for an interlaboratory Study of the Vickers Microindentation Hardness Test for Ferrous Specimens in Diagonal Units (μm)

Specimen	Test Force (gf)	Average Diagonal (μm) $\bar{d}$	Standard Deviation (μm) S_d	Repeatability Standard Deviation (μm) S_r	Reproducibility Standard Deviation (μm) S_R	Repeatability Limit (μm) r	Reproducibility Limit (μm) R
A	25	13.89	0.75	0.30	0.80	0.85	2.24
	50	19.81	0.61	0.34	0.68	0.95	1.91
	100	28.10	0.57	0.45	0.70	1.26	1.96
	300	49.19	0.75	0.72	0.99	2.02	2.77
	500	63.65	0.81	0.88	3.16	2.47	1.13
	1000	90.48	0.98	1.31	1.53	3.66	4.28
B	25	9.35	0.40	0.25	0.46	0.69	1.28
	50	13.06	0.37	0.23	0.42	0.63	1.18
	100	18.51	0.39	0.39	0.52	1.09	1.47
	300	32.11	0.43	0.30	0.50	0.84	1.41
	500	41.68	0.51	0.36	0.60	1.00	1.69
	1000	59.21	0.55	0.52	0.72	1.46	2.03
C	25	10.81	0.53	0.19	0.56	0.54	1.56
	50	15.13	0.42	0.20	0.46	0.57	1.29
	100	21.34	0.40	0.22	0.45	0.62	1.25
	300	36.85	0.38	0.21	0.43	0.59	1.20
	500	47.68	0.55	0.24	0.59	0.67	1.64
	1000	67.60	0.58	0.33	0.65	0.93	1.83
D	100	24.50	0.43	0.29	0.50	0.82	1.40
	300	42.52	0.41	0.28	0.48	0.80	1.35
	500	55.02	0.50	0.25	0.55	0.70	1.54
	1000	78.14	0.70	0.34	0.77	0.97	2.15
E	100	15.61	0.40	0.18	0.43	0.52	1.20
	300	27.25	0.41	0.25	0.46	0.70	1.30
	500	35.26	0.43	0.20	0.46	0.55	1.30
	1000	50.06	0.41	0.24	0.46	0.67	1.29
T	300	23.94	0.47	0.17	0.49	0.49	1.38
	500	31.00	0.51	0.21	0.55	0.59	1.53
	1000	44.12	0.50	0.25	0.55	0.69	1.53

TABLE X3.4 Precision statistics for an interlaboratory Study of the Knoop Microindentation Hardness Test for Ferrous Specimens in Hardness units (HK)

Specimen	Test Force (gf)	Average Diagonal (μm) $\bar{d}$	Standard Deviation (HK) S_a	Repeatability Standard Deviation (HK) S_r	Reproducibility Standard Deviation (HK) S_R	Repeatability Limit (HK) r	Reproducibility Limit (HK) R
A	25	35.61	22.07	11.35	24.29	31.56	68.41
	50	51.77	13.64	11.39	17.05	32.05	47.96
	100	74.84	11.20	12.02	15.49	33.68	43.61
	300	132.28	9.70	9.48	12.91	26.60	36.21
	500	171.51	5.84	6.94	8.52	19.45	23.86
	1000	243.11	3.41	5.86	6.26	16.43	17.52
B	25	23.66	51.07	25.79	55.92	72.09	157.50
	50	34.33	33.07	19.70	37.65	55.27	105.55
	100	49.61	26.11	15.15	29.38	42.45	82.72
	300	88.64	17.04	10.79	19.49	30.04	54.74
	500	115.48	15.52	10.26	18.02	28.75	50.50
	1000	164.38	10.57	9.74	13.71	27.24	38.34
C	25	27.62	44.96	16.55	47.67	46.65	134.05
	50	39.47	26.39	11.57	28.24	32.19	79.67
	100	56.66	16.43	10.01	18.78	28.02	52.50
	300	100.14	10.63	6.89	12.24	19.22	34.29
	500	130.19	9.67	5.35	10.83	15.03	30.26
	1000	184.84	8.07	5.36	9.37	15.01	26.24
D	25	31.04	24.75	10.94	26.42	30.48	74.60
	50	44.64	13.60	7.36	15.20	20.80	42.46
	100	64.22	11.61	7.20	13.33	20.32	37.34
	300	113.94	5.43	4.73	6.67	13.22	19.23
	500	148.16	5.08	3.24	5.62	9.01	16.32
	1000	210.10	6.23	5.03	7.67	14.06	21.49
E	25	20.02	63.88	42.57	74.54	120.56	208.90
	50	29.03	58.20	27.92	63.44	78.02	178.37
	100	42.21	43.53	19.68	46.94	55.28	131.37
	300	76.03	19.43	10.30	21.56	28.76	60.27
	500	99.25	15.43	7.13	16.74	19.94	46.74
	1000	141.67	12.71	8.51	14.81	23.92	41.55
T	25	17.14	124.50	67.85	138.69	191.33	395.07
	50	25.59	87.53	39.91	95.19	112.23	266.90
	100	37.20	80.22	28.75	84.10	80.77	237.05
	300	67.43	38.71	18.10	42.06	50.70	117.74
	500	88.27	22.97	13.65	26.07	38.28	73.09
	1000	126.96	20.44	10.43	22.39	29.07	62.90

TABLE X3.5 Precision statistics for an interlaboratory Study of the Vickers Microindentation Hardness Test for Ferrous Specimens in Hardness units (HV)

Specimen	Test Force (gf)	Average Diagonal (μm) $\bar{d}$	Standard Deviation (HV) S_s	Repeatability Standard Deviation (HV) S_r	Reproducibility Standard Deviation (HV) S_R	Repeatability Limit (HV) r	Reproducibility Limit (HV) R
A	25	13.89	25.99	10.38	27.73	29.46	78.52
	50	19.81	14.56	5.11	16.23	22.69	45.77
	100	28.10	9.53	7.52	11.70	21.08	32.84
	300	49.19	7.01	6.73	9.26	18.90	25.94
	500	63.65	5.83	6.33	22.75	17.78	6.13
	1000	90.48	4.91	6.56	7.66	18.34	21.45
B	25	9.35	45.41	28.37	52.24	78.48	146.56
	50	13.06	30.81	19.15	34.98	52.51	98.63
	100	18.51	22.81	22.81	30.42	63.85	86.24
	300	32.11	14.45	10.06	16.81	28.24	47.43
	500	41.68	13.06	9.22	15.37	25.62	43.32
	1000	59.21	9.83	9.29	12.87	26.09	36.29
C	25	10.81	38.95	13.95	41.16	39.69	115.71
	50	15.13	22.50	10.71	24.64	30.54	69.32
	100	21.34	15.27	8.40	17.18	23.67	47.79
	300	36.85	8.45	4.67	9.56	13.12	26.70
	500	47.68	9.41	4.11	10.09	11.46	28.07
	1000	67.60	6.96	3.96	7.80	11.17	21.98
D	100	24.50	10.85	7.31	12.61	20.69	35.36
	300	42.52	5.93	4.05	6.95	11.38	19.55
	500	55.02	5.57	2.78	6.12	7.79	17.15
	1000	78.14	5.44	2.64	5.99	7.54	16.72
E	100	15.61	39.01	17.55	41.94	50.73	117.35
	300	27.25	22.55	13.75	25.30	38.50	71.56
	500	35.26	18.19	8.46	19.46	23.27	55.03
	1000	50.06	12.12	7.10	13.60	19.81	38.15
T	300	23.94	38.12	13.79	39.74	39.74	112.09
	500	31.00	31.75	13.07	34.24	36.73	95.35
	1000	44.12	21.59	10.80	23.75	29.80	66.11

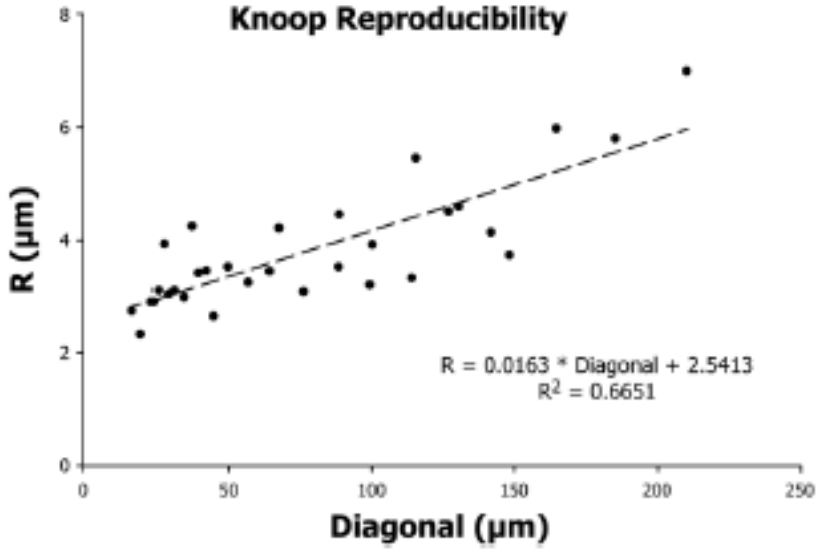


FIG. X3.1 The Relationship between Reproducibility (R) and Diagonal length ($\bar{d}$) from Table X3.1 in μm units, for the Knoop Hardness Tests for Specimens B, C, D, E and T

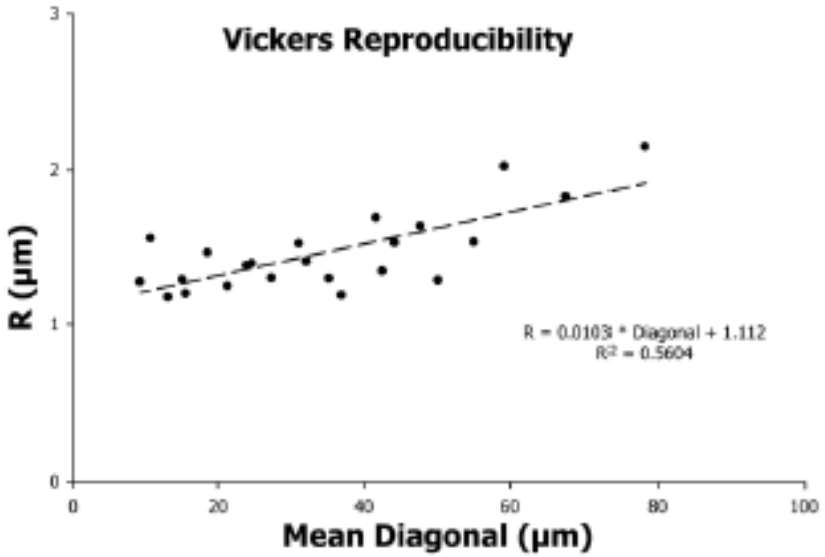


FIG. X3.2 The Relationship between Reproducibility and Diagonal length (*d*) from Table X3.2 in µm units, for the Vickers Hardness Tests for Specimens B, C, D, E and T

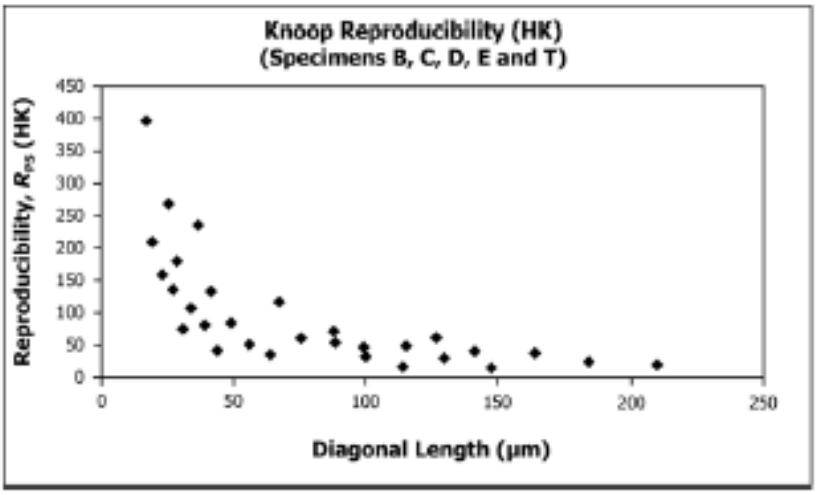


FIG. X3.3 The Relationship between Reproducibility (R) and Diagonal length (*d*) from Table X3.3 in HK units, for the Knoop Hardness Tests for Specimens B, C, D, E and T

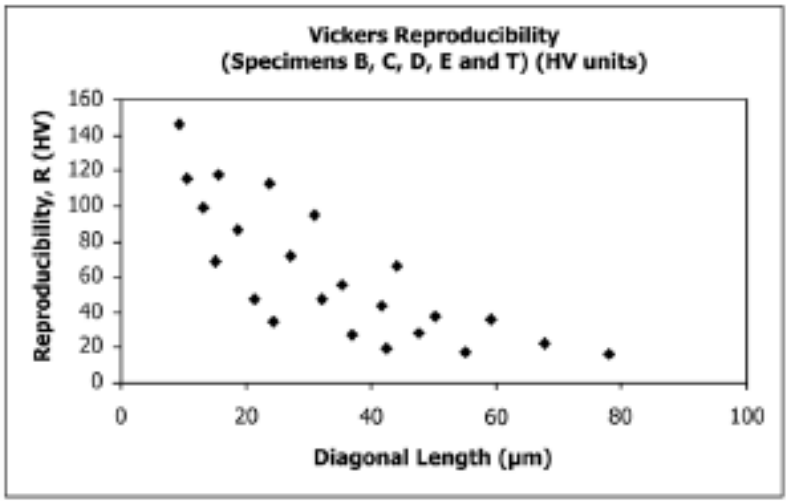


FIG. X3.4 The Relationship between Reproducibility (R) and Diagonal length (d) from Table X3.4 in HV units, for the Vickers Hardness Tests for Specimens B, C, D, E and T

X4. RECOMMENDATIONS FOR LIGHT FORCE MICROINDENTATION HARDNESS TESTING

X4.1 Introduction

X4.1.1 Microindentation hardness of materials can be determined using a variety of loads to force the indenter into the test piece. Testing is considered to be using a light force when the force in use produces indentations with a diagonal length of less than 20 µm. Both Knoop and Vickers hardness numbers increase in proportion to the inverse of the square of the indentation diagonal length, Eq 2 and Eq 7. Thus, hardness numbers obtained from indentations with diagonals measuring less than 20 µm are much more sensitive to variations of a few tenths of a micrometer in the actual or measured length of the diagonals than hardness numbers obtained by measuring larger indentations. Creation of valid indentations, and the accurate measurement of their diagonals, becomes even more imperative as the indentations become smaller. For example, consider a material with a Vickers hardness of 500. For a force of 100 gf, the diagonal length would be 19.258 µm. To maintain an error of ± 1 %, the accuracy of the diagonal measurement must be ≤ 0.096 µm. Similarly, for a material with a Knoop hardness of 500, when tested with a 20 gf force, the diagonal length would be 23.86 µm. To maintain an error of ± 1 %, the accuracy of the diagonal measurement has to be ≤ 0.12 µm. Measurements to this level of accuracy are impossible to achieve by light optical microscopy. Because of the inherent difficulties involved in obtaining and measuring indentations with diagonals less than 20 µm, and the increasing effect of possible indentation or measurement errors, light force microindentation hardness testing requires precautions in addition to those normally necessary. Small indentations may be due to high test piece hardness or the use of light forces, or both. In either case, some of the concerns involved with obtaining accurate hardness results are addressed in this appendix.

X4.2 Scope

X4.2.1 These recommendations provide guidance and suggest additional precautions for microindentation hardness testing when the measured indentation diagonals are less than 20 µm in length.

X4.3 Environment

X4.3.1 *Vibration:*

X4.3.1.1 Vibration of the microindentation hardness tester during a light force test can cause a large percentage increase in the measured diagonals. Reasonable accuracy and precision can only be achieved when the test instrument is isolated from vibration as much as possible during testing. Use of an isolation table or isolation platform is mandatory. Airborne vibrations in the vicinity of the test instrument, such as air currents and loud noises, are to be avoided.

X4.3.1.2 It is recommended that test instruments not be located above the ground floor of the building due to the increase in vibration usually experienced by the upper floors. Test instruments should be located in areas away from machinery that may cause low (<20 Hz) frequency vibrations, since low frequencies are more easily transmitted through isolation tables and platforms.

X4.3.2 *Level*—Microindentation hardness testers must be level in order to obtain usable information. Errors due to minor un-leveling become more important as the forces become lighter.

X4.3.3 *Temperature*—Control of the temperature of the specimen, testing instrumentation, and surrounding area should be considered. It is recommended that these temperatures be maintained at 23 ± 3°C. As the length of the measured

diagonal becomes smaller, it may be necessary to increase control of temperature to reduce variability.

X4.4 Specimens

X4.4.1 *Specimen Preparation:*

X4.4.1.1 Usually, test pieces require mounting. Care must be taken to ensure that the specimens are well supported in the mounting material, and that the surface to be tested can be placed into the test instrument such that it will be normal to both the loading and optical axes.

X4.4.1.2 The surface properties of the test specimen must not be altered due to specimen preparation. Metallographic specimen preparation should be performed using accepted techniques known to eliminate all preparation-induced deformation on the test surface of the specimen. Light etching followed by light re-polishing may be used to further decrease the thickness of any deformed layer. Electropolishing may provide surfaces essentially free of deformation due to preparation when properly performed. Areas to be tested must appear flat in the field of focus of the microscope used to measure the diagonals of the indentations.

X4.4.1.3 The surfaces to be tested should be as clean as possible. Care must be taken to avoid surface contaminants that may be absorbed into the surfaces of some materials such as polymers or ceramics.

X4.4.2 *Microstructure of Specimen*—If the microstructure of the test piece is on the same size scale as the indentation diagonal length, an increase in the variability of the hardness data should be expected. Indentations placed within a single grain will experience resistance to deformation somewhat dependent on the orientation of that grain to the test surface. Since these orientations are normally random, variability of results is increased. Indentation diagonal lengths can vary depending upon the number of grain boundaries traversed by the indentation. Multiphase material systems will provide indentation diagonal lengths that may be proportional to the volume percentage of each phase included within the volume of deformation caused by the indentation. In the above cases, an increase in the number of measurements taken will be necessary to provide meaningful results.

X4.5 Instruments

X4.5.1 *Magnification of Microscope*—Classic microindentation hardness testers make use of optics that usually provide magnifications from 400 to 600×. Higher magnifications are required when performing light force testing. Specimens may be removed from the test instrument following the indentation operation, and the diagonals of the indentations measured using a separate high quality light (or SEM measurements, see X4.7.1) microscope capable of providing higher magnifications.

X4.5.2 *Optical Quality of Microscope*—Use of highly corrected objectives with numerical apertures of 0.9 or greater is recommended. Use of dark field illumination or differential interference contrast may improve the contrast of the image and also enhance the user's ability to detect the ends of the indentations.

X4.5.3 *Diagonal Measuring Device*—The measurement technique and the devices used to perform the measurements should be capable of discerning differences in length of 0.1 m or less. In some cases, it may be preferable to obtain a photomicrograph of the indentation first, and measure the length of the diagonal as seen in the photomicrograph. In all cases, calibration of magnifications and measuring devices is necessary.

X4.5.4 *Accuracy of Forces*—Often, small indentation diagonal lengths are the result of the use of very light forces, in many cases 10 gf or less. Force accuracy of ± 1.5 % is required. For light forces, this requires that no oils, dust, or other minor contaminants be present. For example, when using a force of 2.0 g, contaminants with a total mass of more than 0.02 g render the results of the test invalid.

X4.5.5 *Loading Rates*—When using light forces, the impact of the indenter on the surface of the test piece can cause significant inaccuracies to occur. Use of the slowest loading rate available for each instrument is recommended.

X4.5.6 *Indenters*—Greater repeatability, accuracy, and precision may be obtained by the careful selection of indenters. Verification of the included angles of the faces, the degree of mismatch at the vertex, and the sharpness of the edges are appropriate criteria for the selection of indenters. Using the manufacturer's certification, the exact indenter constant should be calculated and used to minimize errors.

X4.6 Measurement of Indentations

X4.6.1 Indentations that do not appear symmetrical should not be considered valid for diagonal measurement. A difference in symmetry greater than 10 % should be addressed with concern. If consistently asymmetrical indentations are obtained, the alignment of the specimen to the indenter should be adjusted. If the problem persists, the microindentation hardness instrument should be serviced by a qualified technician.

X4.7 Scanning Electron Microscope

X4.7.1 Measurement of indentation diagonals using a scanning electron microscope is possible. However, careful calibration of the SEM photographic image at the exact magnification to be used is essential. For these measurements, the specimen must be perpendicular to the beam, that is, the tilt angle should be 0°. The accelerating voltage and other parameters should remain as they were for calibration. (The SEM should be calibrated in both the *X* and the *Y* directions; refer to Practice E766. Indentations to be measured should not extend to the periphery of the SEM field of view, as the video signal can be distorted at the edges of the video monitor.

X4.8 Video and Automatic Measuring Systems

X4.8.1 Typical video or computerized measuring systems lack the necessary resolution for obtaining acceptable results when indentation diagonal lengths are less than 20 µm. Loss of resolution within the digitized image can cause a substantial decrease in the accuracy of the measurement. Extremely high resolution video cameras and monitors, when appropriately

assembled into a measuring system, may be capable of resolution sufficient to provide accurate results.

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SUMMARY OF CHANGES

Committee E04 has identified the location of selected changes to this standard since the last issue (E384 – 11$^{\varepsilon1}$) that may impact the use of this standard. (Approved February 1, 2016.)

(1) This test method was heavily revised. Changes were made throughout the text.